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FLORIDA INTERNATIONAL UNIVERSITY

Miami, Florida

DOPING AS A POSSIBLE MEANS TO CREATE SUPERCONDUCTIVITY IN GRAPHENE

A dissertation submitted in partial fulfillment of

the requirements for the degree of

DOCTOR OF PHILOSOPHY

in

ELECTRICAL ENGINEERING

by

Kiar J. Holland

2016

To: Interim Dean Ranu Jung College of Engineering and Computing

This dissertation, written by Kiar J. Holland, and entitled Doping as a Possible Means to Create Superconductivity in Graphene, having been approved in respect to style and intellectual content, is referred to you for judgment.

We have read this dissertation and recommend that it be approved.

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Date of Defense: July 6, 2016

The dissertation of Kiar J. Holland is approved.

Interim Dean Ranu Jung College of Engineering and Computing

Andrés G. Gil Vice President for Research and Economic Development and Dean of the University Graduate School

Florida International University, 2016

ABSTRACT OF THE DISSERTATION

DOPING AS A POSSIBLE MEANS TO CREATE SUPERCONDUCTIVITY IN GRAPHENE

by

Kiar J. Holland

Florida International University, 2016

Miami, Florida

Professor Grover Larkins, Major Professor

The possibility of creating superconductivity in Highly Oriented Pyrolytic Graphite (HOPG) by means of doping was investigated. Bulk HOPG samples were doped with phosphorous using either ion-implantation or by Chemical Vapor Deposition growth with phosphine in the gas mixture. The methods for testing the graphene samples, once doped, were done by performing R vs. T measurements, and determining via observation suppressed superconductive characteristics signaling the presence of the Meissner Effect in a strong applied magnetic field. Before doping, the resistance vs. temperature (R vs. T) characteristic of the HOPG was measured. The R vs. T characteristic was again measured after doping, and for surface multilayers of graphene exfoliated from the post doped bulk sample. A 100 to 350 mT magnetic field was supplied, and the R vs. T characteristic was re-measured on a number of samples.

Phosphorous-implanted HOPG samples exhibit deviations from the expected rise in resistance as the temperature is reduced to some point above 100 K. The application of a modest magnetic field reverses this trend. A step in resistance at a temperature of approximately 50-60 K in all of the samples is clearly observed, as well as a second step

at 100-120 K, a third at a temperature range of 150-180 K and a fourth from about 200-240 K. A response consistent with the presence of magnetic field flux pancake vortices in phosphorous implanted HOPG and in phosphorous-doped exfoliated multilayer graphene has been observed. The lack of zero resistance at low temperatures is also consistent with pancake vortex behaviour in the flux-flow regime. The presence of magnetic vortices requires, and is direct evidence of superconductivity.

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ABBREVIATIONS AND ACRONYMS

Highly Oriented Pyrolytic Graphite	HOPG
Yttrium Barium Copper Oxide	YBa ₂ Cu ₃ O _{7-X}
Super Conducting Quantum Interference Device	SQUID

I. INTRODUCTION

Electrical resistance causes a loss of power from point A to point B, due to the effects of Joule heating [1]. This conversion of electrical power leads to heat dissipation. As the temperature increases, the electrical resistance increases further [2]-[14], which can make the device unreliable, inefficient, slow, and increases the probability of premature failure [15]-[41]. In order to offset electrical (resistive) losses in power transmission, utility companies generate more power than is delivered to the end user [1].

Superconductivity is a phenomenon that typically appears at very low temperatures. A superconductor exhibits zero resistance while also expelling a magnetic field [42]. A room temperature superconductive material could facilitate the implementation of many power intensive systems (in MRIs etc.) [43], [44].

A room temperature superconductor would revolutionize technology; not only in the field of electrical and electronic engineering; but also in the extended capabilities attainable and new levels of freedom in the design and uses of electronic devices. Computers would work faster, cooler and more reliably [15]-[41]. Mobile devices would have increased battery life. The cost of electricity generation and transmission would lower, and the list goes on [1].

To date, there have not been any room temperature superconducting materials found [45]-[52]. This is why the search for new types of superconductors with higher critical temperatures is critical.

Carbon is known to possess a large number of allotropes, exhibiting interesting and unique mechanical, chemical and electrical properties [91]. Recently, quite a number of

researchers have turned their attention to graphene [92]-[108], [162], a two dimensional carbon structure that could be used as a test object to study properties necessary for superconductivity [161], [163].

An economical method of obtaining graphene was discovered by Andre Geim and Konstantin Novoselov in 2004 when they pulled ordinary adhesive tape off the surface of graphite [53], [54], producing a monolayer of carbon atoms adhered to the tape. Through their work, they discovered that this single carbon layer was not only the thinnest known material, but also the strongest [53], [54], [111]. The high conductivity and translucent properties of graphene sparked the interest of the photovoltaic industry and touchscreen manufactures, among many other groups. Geim and Novoselov went on to win the Nobel Prize in Physics in 2010 for their work [53], [54].

Doping graphene, due to its close coupling or strong scattering by plasmons as well as phonons has led many to the belief that the Critical Temperature of a superconductor can be raised higher than previously observed in other materials [72]-[78]. More specifically, it has also been suggested that this can be done by raising the density of conduction electrons for each graphene sheet [58]-[66].

Highly Oriented Pyrolytic Graphite (HOPG) was used in preliminary studies of this work, as interlayer coupling in it is weak [109], [110]. This makes it provide a good approximation for graphene. The robust mechanical nature of HOPG, along with its ease of use made it the preferred starting material. An electrical response suggestive of superconductivity in phosphorous (electron donor) implanted Highly Oriented Pyrolytic Graphite was observed. The ultimate critical temperature in this system appeared to be in excess of 100 K. It was noted that the critical temperature may be considerably higher if damage incurred during the doping process was minimized. This further positioned HOPG as the starting material of choice.

The phenomenon described in this work strongly suggests the presence of superconductivity, and as a result, the decision was made to proceed based on the possibility that superconductivity is present. However, it should be noted that it is not possible to rule out some new and previously undiscovered physical manifestation of the extreme anisotropy and two dimensionality of the material. Due to the results of this work, no viable, established alternative to superconductivity can be proposed at this time.

II. PROBLEM STATEMENT

Prior to our work, there has never, to our knowledge, been an exhaustive, systematic attempt to dope graphene to create a high temperature superconductor. Phosphorus doped graphene created by Plasma Enhanced Chemical Vapor Deposition has not been created or studied and should have unique characteristics. A safe, custom system, built in-house, will need to be created to achieve phosphine Plasma Enhanced Chemical Vapor Deposition. We will need to build a custom cryogenic system for measuring graphene and HOPG at near absolute zero while also going to high temperatures so that we do not miss an above room temperature transition. A magnetic field generator will need to be built for the system so that the sample being tested is not disturbed when the physical apparatus is applied. We will need to develop custom software to orchestrate automation from highly accurate voltage measurements, control current generation, compressor, temperature monitoring and temperature controller. A magnetic susceptometer will need to be constructed, capable of near absolute zero

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temperatures while also being sensitive enough to detect slight changes in the magnetic field of a thin film.

Hypothesis:

- Based on our previous work of doping HOPG with boron and phosphorus dopants, we determined that doping using phosphorus and other electron donors could create a high temperature superconductor.
- Graphene doped with electron donors should create flux vortices and become a type II superconductor.
- Graphene doped using plasma enhanced Chemical Vapor Deposition will minimize damage to the lattice and allow longer coherence lengths. This will improve its superconductivity but create flux flow which will add to the resistance.
- Multilayer graphene doped using ion implantation will create columnar defects that will pin the flux vortices and prevent them from moving. This will create pancake vortices and lower the resistance when the graphene is superconducting.
- The mixed state region will have a very wide transition phase since the anisotropy of graphene is much higher than that of known type II superconductors.
- We expect to see a Hall Effect sign reversal since similar type II superconductors see this.

III. BACKGROUND

In doped graphene, it has previously been hypothesized [72]-[74], [76] that the close coupling or strong scattering of electrons by both phonons and plasmons indicates a potential for superconductivity at considerably higher temperatures than previously observed [112]-[136]. Kopelevich *et al.* [57]-[67] reported a few cases of suspected superconductivity in Highly Oriented Pyrolytic Graphite, however the results were inconclusive.

As far as it is known, our prior work [72] is the first in which a systematic attempt was made to substitutionally dope HOPG/graphene/graphite into a superconductive state. Later work by Scheike *et. al.* [140]-[159] and Ballestar [160] also provided hints of possible superconductivity in doped graphite. The work described herein details the efforts in attempting to confirm or disprove these results as originating from superconductivity.

A. Highly Oriented Pyrolytic Graphite (HOPG)

The focus on the use Highly Oriented Pyrolytic Graphite (HOPG) was due to the difficulties involved with doping graphene, and concerns with how to physically handle graphene in a testing environment without damaging samples. HOPG is easily handled and is structurally a "stack" of graphene sheets [55].

HOPG is available in four different grades of quality; with ZYA being the highest and ZYH being the lowest [79]-[82]. The lower the mosaic spread, the lower the angle of deviation of the grain boundaries from the perpendicular axis and hence, a more highly ordered HOPG sample [83], [84]. Thi means that there will also be a larger grain size for higher order HOPG as well [84].

	Grade	Mosaic S	Chin size man	
		Value	Accuracy	Chip size, mm
i i i	ZYA	0.4°	±0.1°	
	ZYB	0.8°	±0.2°	10 10
	ZYD	1.5°	±0.3°	10x10
	ZYH	3.5°	±0.5°	

 Table 1. HOPG Grades. Data Collected and Confirmed from [79]-[84].

While this would affect doping, using lower quality- lower cost ZYH grades for early trials of different doping techniques is more economical. Higher grade HOPG can be used for techniques that prove to be promising in the lower grade ZYH. Furthermore, a slightly less oriented HOPG sample may have beneficial doping properties due to easier implantation in defect areas.



Figure 1: A HOPG sample with scratch used to indicate the bottom side.

B. Type 1 Superconductivity

On July 10, 1908, helium was successfully liquefied by a Dutch physicist named Heike Kamerling Onnes by using several precooling stages as well as the Hampson-Linde cycle [164]. This discovery allowed for testing temperatures that could not be achieved before. By reducing the pressure, he was able to go below helium's boiling point of 4.2 K for a final temperature of 1.5 K [164],[165]. Three years later in 1911, he found that solid mercury wire submerged in liquid helium had a resistance that abruptly dropped to zero at 4.2 K [164],[165]. This was the first discovery of superconductivity [164],[165]. Later, many other single element type I superconductors were discovered, as seen in Table 2.

Material	Tc (K)	Lattice Structure	Material	Tc (K)	Lattice Structure
Lead (Pb)	7.196	FCC	Zinc (Zn)	0.85	HEX
Lanthanum (La)	4.88	HEX	Osmium (Os)	0.66	HEX
Tantalum (Ta)	4.47	BCC	Zirconium (Zr)	0.61	HEX
Mercury (Hg)	4.15	RHL	Americium (Am)	0.60	HEX
Tin (Sn)	3.72	TET	Cadmium (Cd)	0.517	HEX
Indium (In)	3.41	TET	Ruthenium (Ru)	0.49	HEX
Palladium (Pd)	3.3	-	Titanium (Ti)	0.40	HEX
Chromium (Cr)	3	-	Uranium (U)	0.20	ORC
Thallium (Tl)	2.38	HEX	Hafnium (Hf)	0.128	HEX
Rhenium (Re)	1.697	HEX	Iridium (Ir)	0.1125	FCC
Protactinium (Pa)	1.40	TET	Beryllium (Be)	0.023	HEX
Thorium (Th)	1.38	FCC	Tungsten (W)	0.0154	BCC
Aluminum (Al)	1.175	FCC	Platinum (Pt)	0.0019	-
Gallium (Ga)	1.083	ORC	Lithium (Li)	0.0004	BCC
Molybdenum (Mo)	0.915	BCC	Rhodium (Rh)	0.000325	FCC

 Table 2. Type 1 Superconductors. Data from [166].

It can be seen that excellent conductors such as copper, silver and gold are not superconductors [166]. This is because they are in a tightly packed FCC lattice structure that creates damping of the electron phonon interaction [166]. The FCC lattice superconductors that appear on the list are able to create adequate lattice vibrations because they have a low modulus of elasticity which promotes phonon-mediated electron coupling [166].

C. Meissner Effect

In 1933, German physicists Walther Meissner and Robert Ochsenfeld discovered that a superconductor would expel its magnetic field when transitioning to its superconducting state [167]-[171]. This phenomenon that would later be known as the Meissner Effect, means that superconductors are perfect diamagnets in addition to being a perfect conductors [167]-[171]. The superconductor will exclude magnetic fields that would have otherwise flowed through it, by means of current loops to cancel these fields out (screening currents) [167]-[171]. However, this cancelling breaks down when the applied magnetic field crosses a critical value H_c [167]. After crossing the critical value, superconductors, after crossing the critical value, there will be a mixed state (also known as a vortex state) where the magnetic flux will increasingly penetrate the material even though there will remain to be no resistance to electric current [170],[171]. There is then a second critical applied field strength where superconductivity will completely cease [170],[171].

D. Inherent Superconductive Traits

Brothers Fritz and Heinz London showed in 1935 that the magnetic field decays exponentially from the surface, which would later be known as the London penetration depth [172]. Although it provided an explanation for the Meissner effect as well as resistanceless transport through experimental observations, it did not provide microscopic explanations [173], [174]. These microscopic explanations were given in 1957 by BCS theory [175]. In 1956, Leon Cooper described a phenomenon where fermions (such as electrons) can have an arbitrarily small attraction towards each other and lead the pair to have a lower energy than the Fermi energy [174]. This shows that the fermions are paired which became known as Cooper pairing [170], [173]-[176]. This is a unique phenomenon that happens at low temperatures. Normally, electrons would repel each other due to each having a negative charge as stated by Coulomb repulsion [170], [173]-[176]. At low temperatures, the positive ions of the lattice will be attracted to the electrons enough to move toward it and create an area of positive charge density surrounding that area in the lattice [170], [173]-[176]. If the positive charge created is high enough, it will attract other electrons and overcome their repulsion to each other to create a pair [170], [173]-[176]. After this electron-phonon interaction develops a Cooper pair, the result adds the half spin electrons to create a boson [170], [173]-[176].

In 1957, John Bardeen, Leon Cooper, and John Robert Schrieffer found that the condensate of these Cooper pairs could explain, on a microscopic level, superconductivity [170]. Since Cooper pairs become a boson, they can form a large Bose-Einstein condensate with overlapping pairs [170], [173]-[176]. To break these pairs, they would need to break all the pairs that have condensated as a whole, which creates an oppositional barrier and is a crucial necessity for superconductors [170], [173]-[176]. This was called BCS theory (an abbreviation of their last names) and they went on to receive the Nobel Prize in Physics in 1972 [176].

E. Type 2 Superconductors

In 1957, superconductors were classified into two types when Alexei Alexeyevich Abrikosov investigated what happen in Ginzburg-Landau theory if k were large instead of small where k is the ratio between superconducting penetration depths and coherence length [177],[178]. Type II superconductors are comprised mostly of metallic compounds and alloys which can yield a higher critical temperatures T_c than type I superconductors [177],[178]. The superconducting "perovskites" (metal-oxide ceramics that normally have a ratio of 2 metal atoms to every 3 oxygen atoms) also belong to this type II group [177],[178].

The A15 phases are series of intermetallic compounds with the chemical formula $A_{3}B$ (where A is a transition metal and B can be any element) and have a specific structure [179]-[184].

The first time the A15 structure compound was observed was in 1931 when an electrolytically deposited layer of tungsten was examined [179]. Several compounds of the A15 structure were discovered in the following years including the discovery of vanadium silicide (V_3 Si) which exhibited superconductivity at around 17 K in 1953 [179]-[184].

In 1954, Niobium-tin (Nb₃Sn) was discovered to be a superconductor [185],[186]. In 1961, after several years of investigating, this compound showed that it could superconduct with large currents and strong magnetic fields. This gave it practical applications to be used with high-power magnets and electric machinery [185]-[194].

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Niobium and titanium (NbTi) alloy is a type II superconductor with a critical temperature of 10 Kelvins and a critical magnetic field of 15 Teslas [195]-[198]. In 1962, at Atomics International, T.G. Berlincourt and R.R.Hake discovered the high critical magnetic field and high critical supercurrent density that it processes which gives it a critical role in the superconducting magnet industry [199]-[214]. It has been used in all particle accelerators so far and for the vast majority of MRI systems [199]-[214]. It is a ductile alloy with mechanical properties which make it easy to fabricate and use [199]-[214].

The first of the oxide superconductors was created in 1973 by a DuPont researcher named Art Sleight [178]. He found that BaPbO₃ and BaBiO₃ had a critical temperature of 13 K and in the late 1970s, other metal oxides were found to be superconductors as well [178].

The field of superconductivity changed dramatically in 1986, when J. Georg Bednorz and K. Alex Müller produced a material, La₂CuO₄, with a critical temperature of 35 K [215]. This material is different from the previous classes of material in that it has a complex crystal structure made from several components, based around copper oxide units [215]. This discovery earned Bednorz and Müller a Nobel Prize, and was followed a year later by the discovery, by Paul Chu and colleagues, of a superconducting ceramic, YBa₂Cu₃O₇ (often abbreviated to YBCO or 1-2-3 from the ratio of its metal atoms) [215]. This ceramic had an even higher critical temperature of around 92 K [215]. This meant for the first time that a material exhibited superconducting behavior at temperatures above that of liquid nitrogen (77 K), which is much cheaper and easier to handle than liquid helium [215].

In July 30, 1998, Scientists in Rüschlikon, Switzerland succeeded in doubling the transition temperature at which a material becomes superconducting and loses all resistance to the transport of electrical current [216]. In a paper published in "Nature", a group of scientists from IBM's Zurich Research Laboratory, the Universities of Geneva and Neuchatel (Switzerland), as well as Antwerp (Belgium), reported the successful incorporation of strain into the atomic lattice of a superconducting oxide film, thereby raising the transition temperature of the oxide material from 25 to 49 Kelvin [216]. Besides having practical significance and potential for new record transition temperatures, this finding also highlighted the role played by atomic lattice parameters in the mechanism of superconductivity [216].

The theory of type II superconductor and the discovery of superfluidity in helium-3 lead Alexei Alexeyevich Abrikosov, Vitaly Ginzburg, and Anthony James Leggett to be awarded the Nobel Prize in October 7, 2003 [216]. According to their work, vortex lines in a superfluid are analogous to the flux lines that occur in a type II superconductor when it is placed in a magnetic field [216]. In rotating superfluid ³He, the vortex structure is particularly rich [216].

The work of Anthony J. Leggett was crucial for understanding the order parameter structure in the superfluid phases of ³He [216],[217]. His discovery was that several simultaneously broken symmetries can appear in condensed matter [216],[217].

This allowed for a deeper understanding of complex phase transitions in fields such as liquid crystal physics, particle physics and cosmology [217].

In 2015, an article published in Nature by researchers of the Max Planck Institute suggested that under extreme pressure, H₂S transitioned to H₃S and entered a superconductive state with a transition temperature of 203 K [218]. The pressure used was 150 gigapascals which is 1.5 million times atmospheric pressure, in a diamond anvil cell [218]. As of 2015, this gives hydrogen sulfide the highest accepted superconducting critical temperature [218]-[228]. By substituting a small part of sulfur with phosphorus and using even higher pressures than what was used for the 203 K T_c , it has been predicted that it may be possible to raise the critical temperature to above 0 °C and even achieve room-temperature superconductivity [218]-[228]. Their research suggests that other hydrogen compounds could superconduct at up to 260 K which would match up with the original research of Ashcroft [217],[225],[226].

It is important to note that 150 gigapascals is an extremely high pressure to sustain and work with which limits the practicality of using hydrogen sulfide in many applications. Mercury barium calcium copper oxide ($H_gBa_2Ca_2Cu_3O_8$) remains as the highest transition temperature superconductor at ambient pressure that has been confirmed by multiple independent research groups, with a T_c of 133 K [229].

F. Magnetism

Examination of the volume of prior work [232]-[272] done to characterize and model the behaviour of magnetic vortices in layered superconductors leads to the conclusion that pancake vortices would be the preferred vortex form if this extremely anisotropic material (the ratio of the in-plane resistivity to the normal direction resistivity in pure multilayer graphene is greater than 100,000:1) is superconducting [273]-[274]. A more specific conclusion [267], is that for graphene stacks with interlayer distances of 0.2 nm and relatively long magnetic penetration depths (graphene and graphite are diamagnetic materials with high levels of magnetic anisotropy [275], [276]) that the interlayer coupling, if the material was superconductive with a magnetic self-pinning attractive force between pancake vortices in layers *i* and *j* is given as follows:

$$F(\mathbf{r}_{j}, j, i) \approx -\rho(\phi_{j}) \left(\frac{\varphi_{0}}{2\pi\Lambda}\right)^{2} \frac{\sqrt{\rho^{2}_{j}} + i - j^{2} |s^{2} - i - j|s}{\rho_{j}}$$
(1)

where:

$$\Lambda = 2\lambda_{\parallel}^{2} / s \tag{2}$$

A is the two dimensional (2D) thin-film screening length; λ_{\parallel} is the effective penetration depth parallel to the graphene planes and *s* is the interlayer spacing.

Where the layers *i* and *j* are adjacent and the vortices are directly vertically aligned, this reduces to:

$$\mathbf{F}(\mathbf{z},0,1) \approx \hat{z} \frac{\varphi_0^2 s^2}{16\pi^2 \lambda_{\parallel}^4}$$
(3)

It can be seen that that the magnetic pinning force between two pancake vortices in adjacent layers is proportional to the square of the interlayer spacing and inversely proportional to the fourth power of the magnetic field penetration depth within the layer.

The single pancake vortex pinning energy is given by Clem [268] as:

$$U_0 \equiv \left(\phi_0 / 4\pi\right)^2 s / \lambda_{ab}^2 \tag{4}$$

where λ_{ab} is the in-plane penetration depth.

This gives a self-pinning characteristic temperature for a single pancake vortex in $YBa_2Cu_3O_{7-X}$ of 1200 K and vortex motion begins to be a problem at about 1/20 of that temperature. Expressions (5) and (6) below [268] are given for $YBa_2Cu_3O_{7-X}$:

$$U_0 / k_B = 1200 \,\mathrm{K}$$
 (5)

where the flux motion temperature regime is:

$$(U_0 / k_B) / 20 = 60 \text{ K}$$
 (6)

The inter-plane separation of graphene is 0.2 nm and that of YBa₂Cu₃O_{7-X} is 1.2 nm so if the in-plane penetration depth λ_{ab} is the same the expected temperature where flux motion regime begins to be important for graphene would be about 10 K. This is an upper estimate given that the measured conduction anisotropy in graphite is significantly greater than that of YBa₂Cu₃O_{7-X}. It is expected that realistic estimates for the onset temperature for flux-flow would be much lower. Therefore, the expectation is that, even

if the material is superconducting with Cooper pairs, the resistance, due to flux flow effects, would be non-zero even at low temperatures and currents.

The strength of attraction of a pancake vortex to a vacancy due to implantation damage is related to the size of the vacancy and the in-plane penetration depth that governs the physical size of the pancake vortex. The pinning force at low temperatures will not be significant in this highly anisotropic material unless pancake vortices, which can be pinned to a vacancy, have numerous similar vortices pinned at vacancies in adjacent layers. Pancake vortex "stacks" can form at these adjacent vacancies, which may result from implantation damage. The pinning force of these stacks would be from the summation of the vortices in the stack's mutual magnetic and Josephson interactions [266].

The pinning energy and the temperature required to "melt" one of these vortex stacks are proportional. Once a stack melts, all of the vortices in the stack are free to move and immediately contribute to resistive losses in the material. This would lead to the expectation of upwards steps in resistance at the melting temperatures of the various height pancake vortex stacks. Pancake vortex stacks will still form, regardless of the presence of implantation damage; however, the probability of adjacent vacancies leading to the formation of vertical stacks would be less in materials without columnar defects to stack along. A doped-while-grown material would therefore be less likely to have sharp steps as the pancake vortices would be less vertically aligned and more tilted away from the normal to the layers (greater spread in layer-to-layer offsets).

Magnetization measurements on thin film superconductors where the penetration depth is many times greater than the film thickness have been done by Berdiyorov [277]

and a number of theoretical works [275]-[310] that are consistent with the experimentally observed results exist. The results from Berdiyorov are best summarized thus: first, the magnetization is negative and, second, the magnetization has a "valley" or quasi-parabolic shape as a function of temperature and/or applied field. This is quite different from what is seen for thicker, classical, samples where pancake vortices are not formed and the applied magnetic field is expelled from the bulk of the sample.

The primary differences that thin films (where the thickness is far less than the magnetic penetration depth) show in comparison to materials with a unity ratio of thickness to penetration depth (in an AC susceptometer) are: (1) a smaller net signal as the screening is smaller and (2) a broader, depressed, transition as a function of temperature due to the field penetration and vortices [311]-[351].

In Hall effect measurements on mixed state layered superconductors with weak flux pinning, it has been widely observed that the presence of a vortex state with mobile vortices often leads to a sign reversal in the Hall voltage as the sample goes through the transition [352]-[355].

G. Resistance versus Temperature

The Resistance versus Temperature measurement is one of the most basic ways to determine the presence of superconductive properties [415]. Once the critical temperature is passed and the sample goes into the superconducting phase, there will be a sudden drop in resistance [42]-[52], [416].

Prior work on doped HOPG [72] showed that the sample's resistance never went to zero, even at low temperatures. Flux-flow resistance was identified to be the potential reason for non-zero resistance at low temperatures [230], [231]. Flux pinning has been

indicated as the key to reducing this flux-flow resistance in layered high critical temperature semiconductors [230]-[270]. Consideration must also be given to the nanometer scale thickness of the exfoliated graphene film under test, as it relates to the magnitude of the test current used in R vs. T measurements [412]-[414], [417]-[419]. This current must be as small as possible or it could potentially influence the results due to either local magnetic fields or heating.

Due to the sensitivity of carrying out R vs. T measurements on thin films, numerous data points are taken in order for an accurate result to be attained. In order to lessen the difficulty, greater focus should be placed on the ability to detect when even a small portion of the sample is superconducting, which will lead to much smaller steps than seen in classical superconductors.

The four probe measurements are the industry standard for measuring superconductors as well as semiconductors [86]-[88]. For superconductors, the collinear probe arrangement is more popular due to its stronger signal strength at lower resistances, as well as a lower need for the Hall Effect measurement (as with semiconductors) [86]. This helps to increase the signal to noise ratio as well as eliminating the effect of probe resistance or contact resistance [86], [87].

Two outer probes are used to source current while two other probes placed between them measure the voltage drop [86]-[88].



Figure 2: Four probe measurement. Collinear setup. Picture courtesy of [89].

The resistivity is then calculated by using the following:

$$\rho_0 = 2a\pi s \frac{V}{I} = a\rho_0 \tag{7}$$

Where 'a' is the thickness correction factor and s is the spacing between probes, presumed to be equal [89]. Since graphene is extremely thin, the correction factor will be determined by use of the plot below, and substitute it for \mathbf{K} in the following formula, where t is the thickness and \mathbf{m} is the slope:

$$a = K \left(\frac{t}{s}\right)^m \tag{8}$$



Figure 3: Correction factor plot. Picture courtesy of [89].

The delta mode method being implemented by the Keithley Delta Mode System 6221/2182A is displayed below:



Figure 4: Delta Mode reading calculation. Screenshot of Keithley's help menu in test program.

Since the current being supplied is switching polarity but remains at the same absolute value, the difference in potential between V1 and V2 is twice as large as the voltage should be. The same can be said for the difference between V3 and V2. Therefore, we arrive at the expression:

$$V_a = negative _edge = (V_1 - V_2)/2$$
(9)

$$V_b = positive _edge = (V_3 - V_2)/2$$
⁽¹⁰⁾

The final voltage reading would be the average of these two readings:

$$V_f = \left(V_s + V_b\right)/2 \tag{11}$$

$$V_{f} = \left[\left(V_{1} - V_{2} \right) / 2 + \left(V_{3} - V_{2} \right) / 2 \right] / 2$$
(12)

$$V_f = \left[\left(V_1 - V_2 \right) / 2 + \left(V_3 - V_2 \right) \right] / 4 \tag{13}$$

This method is beneficial as it eliminates linear thermoelectric drift. The drift only has to be linear with respect to the 3 points being measured, as seen in the figure below.



Figure 5: Delta Mode Cancelling Thermoelectric drift.

Letting V_t represent the offset caused by the thermoelectric drift and dV_t represent the change in the offset of thermoelectric drift since the initial V_t (to simulate drift going upwards or downwards):

$$V_{a} = \left[\left(V_{1} + V_{t} \right) - \left(V_{2} + V_{t} + dV_{t} \right) \right] / 2$$
(14)

$$V_a = (V_1 - V_2 - dV_t)/2$$
(15)

$$V_{b} = \left[\left(V_{3} + V_{t} + 2dV_{t} \right) - \left(V_{2} + V_{t} + dV_{t} \right) \right] / 2$$
(16)

$$V_{b} = \left(V_{3} + 2dV_{t} - V_{2} - dV_{t}\right)/2 \tag{17}$$

 V_t is removed in expanding V_a and V_b . Once V_f is calculated, we see that all the dV_t s are cancelled:

$$V_f = \left(V_a + V_b\right)/2 \tag{18}$$

$$V_{f} = \left[\left(V_{1} - V_{2} + dV_{t} \right) / 2 + \left(V_{3} + 2dV_{t} - V_{2} - dV_{t} \right) / 2 \right] / 2$$
(19)

$$V_f = \left(V_1 - V_2 - dV_t + V_3 + 2dV_t - V_2 - dV_t\right)/4$$
(20)

$$V_{f} = \left[\left(V_{1} - V_{2} \right) + \left(V_{3} - V_{2} \right) \right] / 4$$
(21)

This final value for V_f represents a single reading that has removed any linear thermoelectric drift that occurred between V_1 and V_3 .

H. Magnetic Field Measurement

To detect the Meissner Effect, a susceptometer (also called a magnetometer) is used to detect the shift in magnetism.


Figure 6: Schematic of magnetometer. Picture courtesy of [90].

M represents the material, s is the coil that is around the material, r is the reference coil, c is the compensation for compensation of imbalance between V_s and V_r with no sample present. p is outer coil generating the magnetic field and G is the current going to the external coil [90]. When V_r and V_s are balanced, (no magnetic field change) V_d should be zero, otherwise the material is causing an imbalance, repelling the magnetic field. To quench the superconducting properties, a strong magnetic field is applied to the sample [387]-[411].

IV. EXPERIMENTAL

All samples used in this work were HOPG ZYH specimens with a mosaic spread of $3.5^{\circ} \pm 1.5^{\circ}$, grain size of 30-40 nm and density of 2.255 - 2.265 g/cm³. Prior to the first use, 10 to 15 monatomic layers were removed from every sample by exfoliation to ensure a pristine layer for initial testing. More than 20 bulk HOPG samples and over 35 exfoliated thin graphite layers, – both doped and non-doped, – were tested in this work. For all samples, preparation proceeded as follows:

All samples were scratched on one side, to indicate the bottom of the sample. Although the HOPG samples have two working sides, it was important to keep track of which side was the bottom after the single sided cleaning and doping process. Before doping, regardless of whether doping was by implantation or through Chemical Vapor Deposition growth, the sample was placed into a closed cycle refrigeration system and the Resistance vs. Temperature (R vs. T) characteristic of the HOPG was measured.

A. Exoliation

Exfoliation, pioneered by Novoselov and Geim [92]-[108], has been used to obtain samples for measurements by a large number of other researchers and is widely utilized.

Kapton[®] tape made of polymide [85] has in the past been used successfully to peel a layer of graphene from highly oriented pyrolytic graphite. This tape is meant for high

vacuum usage due to its uniform silicone adhesive layer which ensures air bubbles will not be trapped in between the tape and the object it is adhering to [85]. Air bubbles will lead to outgassing that can make the vacuum pressure unstable, which is undesirable.



Figure 7: System created for sample preparation. Chuck with through hole to create vacuum, capable of holding down HOPG sample. Silicon Kapton[®] tape was added to give a soft surface for the HOPG to rest, which was found to give a better vacuum seal. Vacuum system with tubes connected to chuck shown on the left.



Figure 8: Sample preparation system with HOPG sample secured to chuck via vacuum seal. A piece of Kapton[®] tape will be laid on top of the HOPG, and carefully pressed down to remove all air bubbles.

This makes Kapton tape ideal for the application of peeling graphene since an air bubble would translate into an area of graphene that may not lift off, leading to a noncontinuous flake of graphene. Also, the resistance of the Kapton[®] tape, in this application, approaches infinity as its resistance is much higher than the resistant measurement devices can read (thus, not contributing to the resistance of the sample being measured).

Theoretically, this process should only remove a single layer of graphite (graphene). Practically however, this is not generally the case. There are many factors causing several layers to be peeled off simultaneously. Examples such as: an uneven sample surface allowing the lowest layers to pull top layers along with it and layers simply being stuck together with a force greater on the top layer than on the bottom. In order to mitigate this effect, a second or even third peel of the initial peel can be taken (thus, dividing the layers of graphene to eventually approach 1).

The move from bulk ion-implanted HOPG to exfoliated (peeled) graphene from ionimplanted HOPG allowed the removal of bulk effects from the physics occurring in the first 20 nm where the greatest damage from the dopant implantation were located [56]. The use of phosphine as a dopant in doped-while-grown plasma Chemical Vapor Deposition films was observed to minimize the implantation damage.

Removal of graphene from tape was investigated using solvents that would dissolve the adhesive. Having graphene without tape would allow for uniform thermal expansion as well as being able to test the doped side directly. It would also allow for closer stacking of samples during susceptibility measurements, which would give a stronger response since the effective sample thickness could be made to be much larger than the London penetration depth.

Each solvent was placed in a glass beaker and graphene on either Kapton[®] or Scotch[®] general purpose tape was placed in the beaker, completely submerged by the solvent. The beaker was then covered with plastic wrap to avoid evaporation and placed in a fume hood for over 24 hours. After that time, the graphene was removed, placed on a lint free towel and removal from the tape was attempted using two tweezers.

Table 3. Solvents tested for graphene lift off from Kapton[®] vacuum tape and general purpose Scotch[®] tape. The green shade denotes lift off while still floating in the solvent. Yellow shading denotes lift off with the aid of tweezers. Red denotes no lift

Solvent	Таре	Lift Off? (y/n)	Difficulty	Time In solvent	Comments	Solubility
3M [®] General Purpose Adhesive Cleaner	Vacuum	Yes	Hard	24hrs	Difficult to get full piece to remove.	
3M [®] General Purpose Adhesive Cleaner	Scotch®	Yes	Hard	24hrs	Difficult to get full piece to remove.	
TCE	Vacuum	Yes	Easy	24hrs	Full lift off in liquid.	ether, ethanol, chloroform
TCE	Scotch®	No	Very Hard	24hrs	Scotch tape curled and could not remove graphene	
Kerosene	Vacuum	Yes	Hard	24hrs	Partial Lift off. Some flakes still remain.	
Kerosene	Scotch®	No	Very Hard	24hrs		
Goof-Off [®]	Vacuum	Yes	Easy	24hrs	Graphene slid off tape when pushed horizontally	
Mineral Spirits	Vacuum	Yes	Easy	27hrs	Graphene stuck to napkin when put upside-down and only used tweezers to lift tape off.	
Mineral Spirits	Scotch®	No	Very Hard	27hrs		
Xylene	Vacuum	Yes	Easy	27hrs	Extremely easy to remove. Graphene stuck to napkin when put upside-down and only used tweezers to lift tape off.	non-polar solvents such as aromatic hydrocarbons
Xylene	Scotch [®]	Yes	Easy	27hrs	Extremely easy to remove. Graphene stuck to napkin when put upside-down and only used tweezers to lift tape off. Easiest scotch tape removal.	
MEK	Vacuum	Yes	Easy	27hrs	As easy as Xylene, Mineral Spirits.	Water
MEK	Scotch®	No	Very Hard	27hrs	Pieces came off but scotch tape was gooey and staying on.	
Goo-Gone [®]	Vacuum	Yes	Easy	27hrs		
Goo-Gone [®]	Scotch®	Yes	Medium	27hrs	Partially stuck to scotch tape but able to get it off in somewhat big flakes.	
Krud-Kutter [®]	Vacuum	Yes	Easy	27hrs		
Krud-Kutter [®]	Scotch®	Yes	Medium	27hrs	Very similar to goo-gone.	
Turpentine	Vacuum	Yes	Easy	27hrs		
Turpentine	Scotch®	Yes	Medium	27hrs		

off.

As seen in Table **3**, TCE, Xylene and MEK were the best solvents from the ones we tested. These either floated off while still in the solvent or were taken off with little to no resistance. Out of those 3, MEK has the added benefit of being soluble in water, making the post removal or residue safe and easy.

For fear of altering the sample, the tape removal process was implemented in later testing. However, this may be an important finding for future work.

B. Resistance versus Temperature Measurement

In order to achieve a high level of sensitivity, there should be little to no noise, little to no thermoelectric error and high precision present in the measuring equipment. The Keithley Delta Mode System 6221/2182A in conjunction with LabView allows for the noise to be minimized, thermoelectric error to be eliminated completely while being able to take measurements at a 1 nV resolution [86].



Figure 9: Keithley 6221 DC and AC current source (on left) and Keithley 2182A nanovoltmeter (on right) with external cooling fan in between to keep airflow constant for cooling of the device.



Figure 10: The peeled sample lifted from the HOPG, loaded into the cryogenic system where probes are placed collinearly onto a continuous area of graphene.

The probes are placed firmly enough to maintain contact, but not so firm that they puncture the material. This is verified after the sample is taken out of the chamber by making sure no holes have been created.

During this measurement portion, the temperature is brought down to 2.5 Kelvin by a compressed helium cooled cryogenic system. This is done under vacuum for the health of the cryogenic system but should not affect the measurements.



Figure 11: Colinear Probes positioned on sample with broken pieces of Lanthanum Aluminate (LaAl) placed near the edges to prevent curling.

After several trial runs of bringing samples down to 2.5 Kelvin and back to room temperature, the ends of the samples were observed to be curling upwards. This curling had a noticeable change on the response of the sample, thus a method to secure the

sample was achieved by placing Lanthanum Aluminate (LaAl) on the edges. LaAl was chosen for its high thermal conductivity, which would ensure that the sample temperature itself would not be affected. LaAl was also chosen due to its low electrical conductivity, which ensured that there would be no shorting or interference between collinear probes.

When signs of superconductivity are present, additional R vs. T measurements is carried out in the presence of a magnetic field. Characteristics that are reduced substantially in the presence of a magnetic field can be interpreted as possibly being due to superconducting properties so it is also important to apply this magnetic field without physically disturbing the sample.



Figure 12: Coil created in-house to generate magnetic field. This slides over the outer shield and was adjusted to make the center of the coils on the same plane as the sample. The ends of the copper coils are connected to a 4 amp DC source.

C. Programming for Resistance versus Temperature Measurements

Extensive programming was done in LabView to efficiently carry out the laborious runs. This was an ongoing process refining and optimization. Additions were made to prevent any previous errors that we saw from happening again. One of the first programs that were created for the R vs. T measurements was the data acquisition VI. This fully automated the Delta measurements as well as monitoring the temperature in the

cryogenic system. The program takes an initial temperature measurement to determine if the run is cooling or warming and then determines if it will need to turn the heaters on later. If it determines that it is cooling, it will take a packet of 250 measurements and then divide them into 10 sub packets. Each of those 10 sub packets will be an average of 25 measurements. For warming, it will take the full packet of 250 measurements and simply get the average to give 1 average measurement from the entire packet. It then displays this information and records it along with other pertinent information, to a Comma Separated Value file before moving on to the next measurement. Comma Separate Value files allow the data to be saved in a table structure while being able to opened by text editors or as spreadsheets. For cooling runs, the program text messages the user to alert them that the cooling run has completed. This lets the user know that they need to turn off the compressor as well as the water that cools the compressor. Then it sends the Comma Separate Value file and the picture of the *R* vs *T* on screen data display to everyone on the team which it also does at the end of the warming run (see Appendix 26 to Appendix 51).

Later, this program was turned to a subVI with an outer VI that controlled the compressor (see Appendix 25 to Appendix 38).



Figure 13: Flow chart for R vs. T measurement subVI for data acquisition.



Figure 14: Flow chart for *R* vs. *T* measurement subVI for data acquisition continued.

All measurements were averaged delta mode in nature and each data point recorded was the result of 250 individual readings averaged together. Measurement speed was approximately 10 seconds per final averaged data point recorded. All measurements were made at the minimum practical stimulus current, always 10 μ A or less (typically 1 μ A), to avoid current induced degradation of the responses. After initial testing, the samples were doped with phosphorous using either ion-implantation or by Chemical Vapor Deposition growth with phosphine in the gas mixture.

D. Doping HOPG

One electron donor (phosphorous), and an electron acceptor (boron) were selected initially for testing the hypothesis. The primary doping method of ion implantation was preferred simply due to expediency. Low energies and doses were used to minimize damage.

A response suggestive of superconductivity in phosphorous (electron donor) implanted HOPG was observed [72]. The estimated critical temperature in this system is in excess of 100 K and, may very likely be considerably higher if damage incurred during the doping could be further minimized. It must be stated that the observed "superconductive type" effect is very likely confined to a very thin layer somewhat further into the HOPG than the peak of the implantation distribution (Figure 15). Doping with electron acceptors (boron) [72] has not been observed to induce the effect despite their probably having caused less damage (lower mass, lower dose and same energy) to the HOPG than the phosphorus.

The computed depth profile of the ion-implanted phosphorous in graphite and the computed damage profile are shown in Figure 15, curves 1 and 2. The corresponding computed profiles for the ion-implanted argon in graphite are shown in Figure 33, curves 3 and 4. Since there is no characterized implantation model for the stopping power of HOPG as a substrate we selected the closest substrate material in the library, graphite, for the simulation.



Figure 15: SRIM simulated distribution of (1) phosphorus ions implanted in graphite at Ep = 10 keV, (2) damage in graphite lattice caused by implant and (3) probable region of effect.

Minimal doping and energy levels were deliberately selected in order to minimize the damage done by the implantation to the graphene sheets in the HOPG. This reduces the disorder in the lattice. This damage could potentially provide scattering centers. These scattering centers would very likely have a negative impact on any electronelectron coupling mechanism, regardless of whether the coupling mechanism is phonon or plasmon mediated.

An array species were chosen for ion-implantation doping to provide us with a bigger picture of what dopants were most promising for investigation. Prior to doping, SRIM simulations were performed to characterize their stopping characteristics seen in Table **4**.

Atom Crystal Atomic Lateral Electron Ionic Valence Projected Longitudinal Element Radius Radius Donor or Straggle Mass Charge Electrons Range (A) Straggle (A) Acceptor? (A) (pm) (pm) (amu) Aluminum 118 67.5 3 +3 27 acceptor 149 49 37 Arsenic 114 72 3 +5 75 donor 104 25 20 Beryllium 112 59 2 +2 9 442 171 130 acceptor Fluorine 42 22 7 +7 19 193 68 50 acceptor 90 7 Lithium 167 1 +1 612 234 187 acceptor Nitrogen 56 30 3+ 5 14 donor 230 84 62 Sodium 190 116 1 +1 23 169 58 43 acceptor 6 32 30 Sulfur 88 51 4 +donor 125 38 Phosphorus 98 5 31 132 32 58 3+ donor 41 71 72 22 17 71 Argon 0+8 40 neutral

Table 4. Stopping and range for ion-implanted species at 10 keV and 5 keV for argon. Green highlights the species that showed features that warrant further investigation.

Each dopant was ion-implanted at a low dose $(1.2 \times 10^8 \text{ cm}^{-2})$ and on a separate HOPG sample with a high dose $(1.2 \times 10^{11} \text{ cm}^{-2})$ using an energy of 10 keV and current of 104 pA. Argon was used to create columnar damage, which will be seen in sample 023

and was implanted at 5 keV to give it a shallower implantation depth than the previous implanted phosphorus.

investigation.						
Element	Low Dose (ions/cm ²)	High Dose (ions/cm ²)	Concentration at Low Dose (atoms/cm ³)	Concentration at High Dose (atoms/cm ³)		
Aluminum	1.20E+08	1.20E+11	9.8E+13	9.8E+16		
Arsenic	1.20E+08	1.20E+11	1.92E+14	1.92E+17		
Beryllium	1.20E+08	1.20E+11	2.81E+13	2.81E+16		
Fluorine	1.20E+08	1.20E+11	7.06E+13	7.06E+16		
Lithium	1.20E+08	1.20E+11	2.05E+13	2.05E+16		
Nitrogen	1.20E+08	1.20E+11	5.71E+13	5.71E+16		
Sodium	1.20E+08	1.20E+11	8.28E+13	8.28E+16		
Sulfur	1.20E+08	1.20E+11	1.26E+14	1.26E+17		
Phosphorus	1.20E+08	1.20E+11	1.17E+14	1.17E+17		
Argon	1.20E+08	None	1.45E+14	None		

Table 5. Dose and concentration for ion-implanted species. All were done at 10 keVwith the exception of argon which was done at 5 keV. All used a current of 104 pA.Green highlights the species that showed features that warrant further

Since phosphorus showed the best results (which will be talked about and shown later in the Results section) and was the easiest for us to work with, many other ion-implantations were performed. One was a multi implantation method that used 5 different energies shown in Table 6. This was performed from highest energy to lowest so that the deepest penetration energy does not have to cross through a prior doped section. These were done with a low dose $(1.2 \times 10^8 \text{ cm}^{-2})$ and current of 104 pA.

Sample	Energy 1 (keV)	Energy 2 (keV)	Energy 3 (keV)	Energy 4 (keV)	Energy 5 (keV)		
065	20	10	6	3	1		

Table 6. Multi-energy implantation of phosphorus for sample 065.

The doped-while-grown material was prepared in a proven graphene Chemical Vapor Deposition system using plasma enhanced Chemical Vapor Deposition on HOPG substrates. The HOPG was used as a seed crystal as it was easier to remove the doped-while-grown layer from the HOPG via exfoliation than a monolayer from copper foil. The gas mixture was methane at 20 sccm, hydrogen at 10 sccm, argon at 14 sccm and 0.1% phosphine in an argon carrier gas at 5 sccm. The plasma power was 30 W for 15 minutes as seen in Table **7**. The substrate temperature was 800 °C and two pieces of partially overlapping copper film were placed adjacent to the HOPG in order to provide a visual witness that graphene had been grown as seen in Figure 16.

 Table 7. Plasma Enhanced CVD recipe.
 Phosphine used was 1000 ppm in argon.

Flow Rate (sccm)				Plasma	Deposition	Deposition	Annealing
Methane	Hydrogen	Argon	Phosphine	Power (W)	Time (s)	Temperature (°C)	Temperature (°C)
20	10	14	5	30	900	800	900



Figure 16: Copper witness visually showing graphene after successful Chemical Vapor Deposition process.



Figure 17: Graphite sample holder with array of HOPG samples loaded. Two copper witness samples are placed at the upper left corner of the array to indicate the position of the first sample in the series.

After doping, either by implantation or through growth, the sample was placed in the closed cycle refrigeration system again. The R vs. T characteristic of the HOPG was then re-measured. All measurements were averaged delta mode in nature and each data

point recorded was the result of 250 individual measurements averaged together. Measurement speed is approximately 10 seconds per final averaged data point recorded. All measurements were made at the minimum practical stimulus current, always 10 μ A or less (typically 1 μ A), to avoid current induced degradation of the responses.

After the measuring of the R vs. T characteristic of the post doped bulk sample the doped surface was exfoliated using silicon adhesive Kapton[®] film tape to remove multilayers of graphene for testing. These multilayer graphene films, still on the tape, were then placed into the closed cycle refrigeration system and the R vs. T characteristic of the graphene multilayer stack was measured. To check that the graphene exfoliations were affected by an applied magnetic field similarly to the bulk phosphorous ionimplanted HOPG shown in Figure 22 a modest, calculated, 100 to 350 milli-Tesla magnetic field was supplied by a dc driven coil placed externally over the refrigerator vacuum shroud and the R vs. T characteristic was re-measured on a number of samples. In addition to the R vs. T measurements, several samples were tested in SQUID magnetometer and AC susceptometer. The results of these measurements and details of Hall effect measurements on one of our exfoliated doped-while-grown samples and a Raman characterization of the film are presented in the results section below. Estimating the thickness of graphene can be roughly done by measuring the light transmission of a monolayer of the material. As a guide, the thickness for of the material can be determined as $T = (.977)^n$ for n layers. Other considerations, such as the film tape transmission, must be made which adds more uncertainty to the estimation method. In order to obtain more accurate information as to the thickness of the exfoliated layers bound to the adhesive backing of the tape, Raman spectroscopy was performed. This was

done at both room temperature and T = 77 K on different positions on the exfoliated material. Room temperature van der Pauw and Hall measurement were performed in an attempt to determine the resistivity of the Phosphine-doped Graphene; as well as the n_{2D} density and mobility values of the material.

E. Susceptometer



Figure 18: Susceptometer System.



Figure 19: Outer coil, inner coil and 2 pieces of single bore alumina.

In Figure 19 we see coils used for susceptometer. On the left, is the outer coil that is responsible for delivering the magnetic field. All turns are in the same direction. Inner coil, on the right, is used for detecting the magnetic field. These have half the windings going in one direction, and the other half going in the opposite direction. This cancels out the field being received and should give a remaining voltage representative of the Meissner effect experienced by the sample. Leads exit on the top.

The Alumina was chosen for its high thermal conductivity, while also being an electric insulator. This ensures that the sample is lowered to the appropriate temperature while also preventing shunting of superconductivity. The diameter allows them to fit into the inner coil and rest the sample between the two pieces of alumina. The position of the sample is adjusted so that it is located in the center of one of the winding directions (so

located either ¹/₄ or ³/₄ of the height of the inner coil). The Alumina is raised and lowered accordingly, using a spacer.



Figure 20: Brass guides, custom made to center coils. These are screwed on top of the coldhead.



Figure 21: Outer coil, inner coil, alumina with sample between alumina pieces and centered using custom sample holder are placed on top of the coldhead and held in placed by 4 brass guides that wedge the assembly in place.

The complete assembly was covered by an aluminum shroud that was wrapped 6 times in aluminized mylar. The final aluminum shield was placed over this to create the vacuum seal. A Signal Recovery Model 7270 DSP Lock-in amplifier was used to create the signal to the outer coil as well as measure using the inner coil. LabView software was created to control all functions of this system (see Appendix 25 to Appendix 37).

To see if the system was working correctly, we used a $YBa_2Cu_3O_{7-X}$ Hairpin sample since this is a known superconductor. We found that a reliable reading could not be yielded, after numerous attempts to balance the coils and find the lowest noise frequency.

V. RESULTS

Phosphorous-implanted HOPG samples were shown in prior work [72] to have exhibited deviations from the expected rise in resistance as the temperature is reduced to some point above 100 K (Figure 22). The relatively large drop in resistance at lower temperatures (by a factor of more than 2) was also considered a possible indication of superconductivity in the sample. It was also noted that the application of a modest magnetic field reversed this trend. All of this as well as the resistance vs. temperature curve for non-doped HOPG are shown in Figure 22 overleaf.



Figure 22: Measured *R* vs. *T* dependence of a phosphorous-implanted HOPG sample. Curve 1 (black) is before implantation, curve 2 (red) is of the phosphorousimplanted sample before magnetic field was applied, curve 3 (green) is of the phosphorous-implanted sample with magnetic field applied and curve 4 (blue) is of the phosphorous-implanted sample after the magnetic field applied in curve 3 magnetic field was removed.

The lack of zero resistance and the modest magnetic field (maximum attainable was less than 0.035 T) required to quench the effect even in the exfoliated multilayer graphene samples is shown in for a representative sample.



Figure 23: *R* vs. *T* of thin film exfoliated from phosphorous-doped HOPG measured without (1) and with 0.035 T applied magnetic field (2).



Figure 24. Aluminum ion-implanted sample. Bulk HOPG with low dose.



Figure 25. Arsenic ion-implanted sample. Peel with low dose.



Figure 26. Beryllium ion-implanted sample. Bulk with low dose.



Figure 27. Fluorine ion-implanted sample. Bulk with low dose.



Figure 28. Lithium ion-implanted sample. Bulk with low dose.



Figure 29. Nitrogen ion-implanted sample. Bulk with low dose.



Figure 30. Sodium ion-implanted sample. Bulk with low dose.



Figure 31. Sulfur ion-implanted sample. Bulk with low dose.

Beryllium, fluorine, lithium and sulfur did not show any features at high or low doses (see Appendix 15 - Appendix 20, Appendix 23, Appendix 24). Aluminum showed features for both low and high while sodium showed features only at high doses (see Appendix 7 - Appendix 9, Appendix 21, Appendix 22). All of the pentavalent species (arsenic, nitrogen, phosphorus) showed features (Appendix 5, Appendix 6, Appendix 10 - Appendix 14). Since phosphorus showed the strongest features and was also the easiest to work with, more in depth research was focused on it.

In order to better understand the potential causes for the observed results, a number of R vs. T characteristics are examined, shown in Figure 32, of similarly exfoliated graphene films taken from bulk phosphorous-implanted HOPG samples.

Comparing characteristics (a)-(d) in Figure 32 it is clear that there is a step in resistance at a temperature of approximately 50-60 K in all of the samples. Upon close examination of the data, it can be determined that there is a second resistance step at 100-120 K, a third at a temperature range of approximately 150-180 K and, yet a fourth at a temperature from about 200-240 K.

Additional steps can be observed in the R vs. T characteristics of just about all of the samples. The most probable explanation is that the features are due to magnetic vortex lattice melting and subsequent flux-flow losses.



Figure 32: Measured *R* vs. *T* of four thin films exfoliated from phosphorousimplanted (Ep = 10 keV, dose 1.2×108 cm-2) HOPG samples. (a) HOPG-008, layer 3; (b) HOPG-019, layer 3; (c) HOPG-019, layer 6; (d) HOPG-021, layer 7. The layer numbers indicate the number of multilayers peeled from the host sample, i.e. layer 7 would be the 7th layer exfoliated from that sample.

To see if additional lattice damage by neutral ion species could increase pinning, which could only occur if magnetic vortices were present, and reduce losses, a sample which had been previously implanted with phosphorous but had not yet been exfoliated was sent back for implantation with argon. This implantation was done at reduced energy (5 keV) and the same dose as the phosphorous implantation $(1.2 \times 10^8 \text{ cm}^{-2})$ to place the

damage in front of the peak in the phosphorous distribution. If the resistance was indeed due to flux-flow, a stronger pinning would be observed in the R vs. T characteristic. Figure 33 shows the computed range distributions for the phosphorous (1) and the argon (3) implants in this HOPG sample and the computed damage distributions caused by implanted phosphorous (2) and argon (4).



Figure 33: This represents the simulated normalized distributions of implanted atoms and lattice damage caused by implanted atoms versus depth in HOPG. This represents the simulated normalized distributions of implanted atoms and lattice damage caused by implanted atoms versus depth in HOPG. Curve 1 is the calculated distribution of the implanted phosphorous, curve 2 is the computed damage caused by the implanted phosphorous, curve 3 is the calculated distribution of the implanted argon and curve 4 is the computed damage caused by the implanted argon.

The R vs. T characteristic of an exfoliated graphene multilayer from this doubly implanted sample (phosphorous followed by argon) is shown in curve 1 of Figure 34. Note that the first R vs. T characteristic taken showed the same qualitative behaviour as the samples in Figure 32.

An anomaly in the form of a small notch can be observed in curve 1 of Figure 34 overleaf. Its size (18 data points each from 250 averaged measurements) is considered too great to be either noise or a measurement error. This notch is at a temperature of 132 K and gave cause to re-measure the sample multiple times. These re-measurements were performed without disturbing the sample or altering the refrigerated environment. In these subsequent R vs. T re-measurements, shown as curves 2 through 4 in Figure 34, a large and abrupt step was observed. The step was in the temperature region of 210-230 K on the first re-measurement. On a second re-measurement it was observed to have moved upward to a temperature of 250-260 K. On a third, and final, re-measurement, the step was noted to have migrated upwards to a temperature of 264-267 K.



Figure 34: *R* vs. *T* of a thin film sample peeled off phosphorous-implanted and then argon-implanted bulk HOPG-023. Curves 1 to 4 are four identical sequential runs with the same probe position.

It can be determined that there is no contact issue present when all four R vs. T characteristics for the graphene multilayer from sample HOPG-023 are plotted together on the same graph (Figure 34). It can be seen that the low temperature and the high temperature resistances have not been changed significantly from run to run. It is also clear that the notch observed in the first R vs. T characteristic is an attempt for the material to move to the resistance state achieved in the later runs and cannot be dismissed as spurious.


Figure 35. Multi energy implantation Sample 065.

For our multi energy ion-implantation sample seen in Figure 35, we see prominent valleys at around 260 K for all runs except the initial run. This valley first appears when the magnetic field is applied and continues to grow after the field is removed for the following runs. On the fourth run, this valley continues all the way down to 200 K. This feature is similar to the phosphorus followed by argon multi energy implantation in Figure 34.



Figure 36. PECVD graphene grown in the presence of phosphine. First peel taken from bulk HOPG.

In Figure 36 we see a representative R vs. T measurement of a graphene grown in the presence of phosphine using our custom plasma enhanced CVD system. The transition can clearly be seen at 240 K and leveling off at 150 K before dropping again at 50 K. As expected, since the flux vortices are not pinned, they are free to flow, rather than being abruptly dismantled as see in the ion-implanted samples. This transition has been seen in over 50 of our samples.

The SQUID magnetometer measurements of a doped-while-grown exfoliated thin film are shown in Figure 37. They consist of a Zero-Field Cooled magnetization run followed by a Field Cooled magnetization run. The hysteresis loop in the Zero-Field Cooled to Field Cooled curves begins to open at a temperature of approximately 260 K.



Figure 37: Magnetization measurements: Zero-Field Cooled (ZFC) and Field Cooled (FC) M/H plots along with a 1 T Field Cooled M/H plot for a film exfoliated from a doped-while-grown Plasma Chemical Vapor Deposition sample.

Additionally, when the horizontal axis of the Zero-Field Cooled magnetization run is converted from T to H/H_{C2} (temperature to the Magnetic Field / Upper critical field of the vortex state) and the data is re-plotted, similar results are attained to those seen in the experiments and calculations done by Berdiyorov's [277] and Novosolev [278]-[287] The Figure 38 (Fig 3.2 in [277]) shows the qualitative shape of a pancake vortex array in a type I superconductor. The thickness here is much less than the magnetic penetration

depth (ratio less than unity). Figure 38(d) would provide the greatest similarity to the samples used in this work as it has no "hard pinning" sites (anti-dots).



Figure 38: The free energy (a-c) and magnetization (d-f) as a function of the applied magnetic field for a filled square (a,d) and for the square with two (b,e) and four (c,f) anti-dots. The insets show the free energy for higher vorticity. The vertical lines show the ground state transitions between different vortex states and open circles indicate continuous transitions between different vortex states. The GL parameter is k = 0.28 [277].

Re-plotting the data of the phosphorous-doped-while-grown Chemical Vapor Deposition exfoliated graphene film Zero-Field Cooled curve given in Figure 37, a comparison can be made with Figure 38(d). The temperature information was converted to the ratio of H to H_{C2} so as to make the comparison with Figure 38(d) [277]. The best comparison that can be made qualitatively is via observing the shape of the curves. This is due to the fact that there is no known value for H_{C2} for the phosphorous-doped-whilegrown Chemical Vapor Deposition exfoliated graphene film. This is shown in Figure 39. Note the step in the curve at low H/H_{C2} in Figure 39. This corresponds qualitatively to one of the discontinuous jumps in Berdiyorov's data and, should the measurement temperature have been taken to lower values, would most likely have continued vertically.



Figure 39: Phosphorous-doped during growth in plasma Chemical Vapor Deposition graphene sample zero-field-cooled magnetization results plotted on same axis type as used in Berdiyorov's work. Note same general shape as in Figure 38(d).

The AC susceptometer measurements for a representative doped-while-grown exfoliated thin film are shown in Figure 40(a) and Figure 40(b). In addition to each of the full-scale plots, a corresponding plot with a magnified vertical axis is shown. A representative plot from a known superconductor is also included as Figure 41 for comparison. Note the small transition that is relatively broad and begins in the area of 150 K (this may be depressed as a result of the measurement field).



Figure 40: AC Susceptometer results for a graphene film on Kapton[®] tape exfoliated from a phosphorous-doped-while-grown Chemical Vapor Deposition sample. (a) film was cut into multiple pieces and the pieces were stacked to increase the screening fraction; (b) magnified vertical axis AC susceptometer results for the same sample and run as shown in (a).



Figure 41: AC Susceptometer results for a Pr2-xCexCuO4 superconductor in the same instrument as was used to produce the data shown in Figure 40. The magnified transition region is shown in the inset.

The final measurements that have been done included a Raman spectrum of one of our exfoliated films to estimate the film's thickness (Figure 42).



Figure 42: Raman spectra for a film on Kapton[®] tape exfoliated from a phosphorous-doped-while-grown Chemical Vapor Deposition graphene sample. The peak ratios give a thickness of approximately 5 monoatomic layers.

The number of layers can be determined by analysing the ratio G/2D. The Raman spectrum plot in Figure 42 indicates a G/2D ratio of 646 K/385 K, which is a little less than 2. This clearly indicates that the graphene material is multi-layered. As the G/2D ratio for 5 monolayers of graphene is about 3.8, the measurements suggest that the exfoliated layers are composed of about 5 to 6 monolayers.

Hall Effect measurements at temperatures of 297 K and 80 K are shown in Figure 43. The change in sign of the Hall voltage is not due to issues in instrumentation. The contacts and extraction calculations were left unaltered between both runs.



Figure 43: Hall effect at T = 293 K and T = 80 K for the same film on Kapton[®] tape exfoliated from a phosphorous doped-while-grown Chemical Vapor Deposition graphene sample as is shown in the Raman spectrum in Figure 42.

For both positive and negative field directions, four different configurations were averaged in resistivity mode and Hall configurations via use of a Hall card and switch from a Keithley model 7001 Switch System.

The great amount of coupling in the conductivity tensor (V_{xx} and V_{xy}) required the data to be symmetrized in order to yield only the induced magnetic field. This is shown in Figure 44. (i.e. $[V_{xy_+B} - V_{xy_-B}]$)



Figure 44: Graph of the four point resistance (averaged over all contact configurations) of the device at room temperature and in 0T field. Vxx preamplifier reversed the polarity. 4 terminal resistance of the film determined to be 45 mOhm.

The Hall signal shown below in Figure 45 (red trace in Figure 43) suggests a two dimensional carrier density of 4.70 X 10^{17} /cm². This result was seen after the different voltage probe and current configurations were made symmetric at room temperature. Using the resistivity obtained from the data shown in Figure 44, an effective mobility of 590 cm²/Vs was calculated. This result is dependent on knowing the exact sample thickness which is somewhat uncertain due to roughness and topological factors.



Figure 45: Graph of the Hall signal after symmetrization the different voltage probe and current configurations were made symmetric at room temperature.

Figure 46 (blue trace in Figure 43) shows the graph of the Hall voltage at 80 K taken after symmetrisation. As can be seen, the density (Hall slope) has not changed dramatically, but increased slightly.



Figure 46: Graph of the Hall signal after symmetrization the different voltage probe and current configurations were made symmetric at 80K.

VI. DISCUSSION

To verify that our delta measurement cryogenic system was working correctly, we did several R vs. T measurements using known superconductors. Since we know what the output should look like for the R vs. T measurement, it would be easy to deduce what problems we were having with our system, if any. The first superconductor that we tested was a YBa₂Cu₃O_{7-X} target that was grown in-house, several years prior. The thickness of these targets was 3 mm while being 8 mm in diameter. Since the volume

was large, we expected to see a clear response. A current bias of 1mA was used after trying several biases out to see which gave the cleanest signal while still giving us a high level of sensitivity. Since the delta resolution decreases a decade for every decade increase in current bias, keeping the current bias low was the key. Lanthanum Aluminate was placed under the YBa2Cu3O7-X target to prevent shunting of superconductivity while also being thermally conductive. Our collinear probe setup was then positioned on the sample, using the same positioning and spacing that we would be using for our HOPG samples. Due to YBa₂Cu₃O_{7-X}'s rough surface, Indium dots were placed between the probes and YBa₂Cu₃O_{7-X} surface. Since Indium is an extremely malleable material it was able to enhance contact when pushed into the surface of the YBa₂Cu₃O_{7-X}. Because the probe spacing is so much greater than the coherence length of YBa₂Cu₃O_{7-X}, the addition of Indium would not affect the superconductive behavior [137]-[139]. On cooling we see that it starts transitioning at 21 Kelvin, then reaching zero resistance at 10.6 Kelvin (see R vs. T for YBa2Cu3O7-X Target in Appendix 1). On warming, it starts transitioning at 88 Kelvin, and reaches zero resistance at 68 Kelvin. This is exactly what we expect to see from a sample of this thickness. The difference in the transition temperature for cooling and warming are largely due to the volume of the sample. The top surface of the target will take longer to cool than the thermocouple under the stage, where the temperature readings are taken. These are the transition temperatures we expected, so we know that our temperature measurements as well as cooling system are working correctly. There's also a lack of noise in this measurement, which is ideal. These are the transition temperatures we expected, so we know that our temperature measurements as well as cooling system are working correctly.

The YBa₂Cu₃O_{7-x} Hairpin filter is 1 millimeter thick and 8 millimeters square. Lanthanum aluminate was placed under the sample in the same manner as the YBa₂Cu₃O_{7-x} target aforementioned, as well as Indium dots being placed under the probes to create better contact. The Hairpin filter has a higher density than the target, with a smoother surface. For the *R* vs. *T* measurement, we see that the Hairpin sample experiences a transition on cooling of 42 Kelvin (see R vs. T for YBa2Cu3O7-X Hairpin in Appendix 2). It reaches zero resistance at 27 Kelvin. For Warming, it begins the transition at 92 Kelvin, and reaches zero resistance at 83 Kelvin. The transition temperature between warming and cooling has a difference of 50 Kelvin whereas for YBa₂Cu₃O_{7-X} Target, the difference was 67 Kelvin. This makes sense because as a sample gets thinner, the transition temperature will be closer for Warming and Cooling since the sample is cooling at a faster rate on the surface. The current bias for the Hairpin filter was 1 micro amp. Since the current was far lower for this measurement, we see a result that is noisier than the Target, which used 1000 times more current.

To further verify our system, we performed an R vs. T measurement using Niobium which is an element type II superconductor (see R vs. T for Niobium in Appendix 3). We used the same set of procedures as the YBa₂Cu₃O_{7-X} Target and Hairpin samples, with the exception of using the Indium between the probes. This was not needed due to the smoother surface of the Niobium. A bias of 1mA was used during cooling but had slightly more noise than desired, so the bias was raised to 10mA for warming. We see that the transition temperature is 9.244 Kelvin. We see an abrupt transition with only 1 data point between the initial transitioning temperature to its zero resistance state. Moreover, this in-between point is most likely due to the transitioning taking place

during the 11 second measuring cycle and being averaged with the resistance before the transition period. Since Niobium has a transition temperature of 9.3 Kelvin, our measurement is only 0.056 Kelvin off. It is also worth noting that the warming run ended at 200 Kelvin due to a power failure. This was far beyond the transition temperature, so the run was not repeated. However, this inspired us to buy a large universal power supply to prevent this from happening in future runs.

During an R vs. T run on a bulk sample, we accidentally discovered what a loss of contact of our probes would look like during a measurement (see Loss of Contact in Appendix 4). The probes were placed on the sample with little to no pressure applied. This meant that there was very little contact being made to the surface of the HOPG at room temperature. During the cooling cycle, thermal contraction would cause the HOPG and lanthanum aluminate to get slightly smaller, resulting in a loss of contact mid run. This occurred at 146 Kelvin and then happened again on warming at 223 Kelvin. Since this is the bulk HOPG sample, it's expected to have a shift between warming and cooling since the top of the sample will not cool as fast, but we see drop to zero resistance instantaneously. This is indicative of a type 1 superconductor, and we know that if HOPG was to superconductive, it would be a type 2 superconductor. There was no noticeable change that we could see, caused by applying a magnetic field. During the cooling run, we watched it the entire time, and noticed that the indicator light at the bottom right-hand corner of the Keithley Model 6221 current source started to blink when it fell to the 0 resistance reading. According to the Keithley Model 6221 manual, this indicates that there is an open circuit. We performed a continuity test via the Triaxial connection on the chamber which connects to the current probes inside to verify this.

Although this was accidental, this gave us a valuable look at what it is like to have a loss of contact during a run. Extra care was given in future runs to make ensure that firm pressure was applied to the probes for ample contact to the sample surface.

Arsenic is stripped of its 3 outermost electrons in the S and P orbitals, prior to ion implantation, to give it a crystal ionic radius of 72 pm. On impact with the HOPG surface, the charge neutralization process returns the electrons, giving it an atomic radius of 114 pm. At this radius, with an energy of 10 keV, the projected range is 104 Å with a longitudinal straggle of 25 Å and lateral straggle of 20 Å. When this pentavalent atom comes to rest, it becomes an electron donor for neighboring carbon atoms since it has 1 extra valence electron.

For the first run on the arsenic peel 001 which had a low dose implantation which yielded a concentration of 1.92E+14 /cm³, there is an abrupt change in slope at 255 Kelvin, which continues to 148.77 Kelvin and levels off until reaching 94 Kelvin (see As Low Peel in Appendix 5. Directly after this run, and without taking the sample out of the chamber or disturbing the probes, we did another run using a magnetic field.

For the magnetic field run, we see that the slope between 94 Kelvin and 255 Kelvin is not as prominent. This is very subtle, and it is not clear if this is experiencing a transition due to flux flow.

For the high dose implantation which yielded a concentration in HOPG of 1.92E+17 /cm³, we see a negative temperature coefficient down to 134 Kelvin (see As Hi Peel in Appendix 6). This phenomenon is seen in semiconductors and means that as the temperature increases, the number of active charge carriers increases. However, since we do not see a freeze out region, this would not be considered an intrinsic or even extrinsic

semiconductor. There does not seem to be any sign of superconductivity at this concentration.

Aluminum is stripped of its 3 outermost electrons in the S and P orbitals, prior to ion implantation, to give it a crystal ionic radius of 67.5 pm. On impact with the HOPG surface, the charge neutralization process returns the electrons, giving it an atomic radius of 118 pm. At this radius, with an energy of 10 keV, the projected range is 149 Å with a longitudinal straggle of 49 Å and lateral straggle of 37 Å. When this trivalent atom comes to rest, it becomes an electron acceptor for neighboring carbon atoms since it has 1 less valence electron.

For Run 002 of our low dose implantation which had a concentration in HOPG of 9.8E+13 /cm³, we see a decrease in resistance from 264 Kelvin down to 210 Kelvin (see Al Low Bulk Run 002 and Run 003 - MF in Appendix 7). This step appears to be a transition indicative of a type II superconductor. The resistance flattens until reaching 114 Kelvin where resistance descends again until reaching the bottom temperature. When a magnetic field was applied, the transition was suppressed by 14 Kelvin for the first transition temperature (250 Kelvin) and by 9 kelvin for the end of that transition (201 Kelvin). After that, the resistance flattens out to 114 Kelvin, which is identical to the previous temperature. When the magnetic field was removed, the first transition is identical to the magnetic field run, beginning at 250 Kelvin and ending at 201 Kelvin (see Al Low Bulk Run 003-MF and Run 004 in Appendix 8). The resistance flattens out for a longer range of temperatures, decreasing after 68 Kelvin.

We see a slightly negative temperature coefficient with the resistance remaining constant from 230 Kelvin to 183 Kelvin in our high dose Aluminum implantation with a

concentration of 9.8E+16 /cm³ (see Al High in Appendix 9). This resembles the 49 Kelvin window for the first transition seen in the low concentration sample, but shifted down 20 Kelvin. This shows that the lower concentration of aluminum ion implantation promoted features consistent with type II superconductors better than the higher concentration.

Nitrogen is stripped of its 3 outermost electrons in the S and P orbitals, prior to ion implantation, to give it a crystal ionic radius of 30 pm. On impact with the HOPG surface, the charge neutralization process returns the electrons, giving it an atomic radius of 56 pm. At this radius, with an energy of 10 keV, the projected range is 230 Å with a longitudinal straggle of 84 Å and lateral straggle of 62 Å. When this pentavalent atom comes to rest, it becomes an electron donor for neighboring carbon atoms since it has 1 extra valence electron.

For our low dose implantation with a concentration in HOPG of 5.71E+13 /cm³, we see a transition at 283 Kelvin that ends at 261 Kelvin (see N Low, Peel 000, Run 002 and Run 003 – MF in Appendix 10). When the magnetic field was applied, although the transition ended at 261 Kelvin as well, we see it diverge from the previous pattern at 278 Kelvin. Instead of having a rounded apex, there appears to be a small amount of suppression present. Afterwards, the two trend lines converge. When the magnetic field run was followed by a non-magnetic field run, we see that the trend lines are very similar, and in some places, overlapping (see N Low, Peel 000, Run 003 – MF and Run 004 in Appendix 11. This may be due to residual flux vortices that were induced by the magnetic field still being present in the sample. This prompted us to remove graphene peels to explore the two-dimensional effect that the nitrogen low concentration doping

may have been having. On the third exfoliation, we see that the first abrupt resistance decrease occurs at 329 Kelvin and ends at 319 Kelvin (see N Low, Peel 003, Run 002 and Run 003 – MF in Appendix 12). From 319 Kelvin to 280 Kelvin, there is a linear decrease in resistance. At 280 Kelvin, there is a linear increase in resistance to 235 Kelvin at which point it begins to decrease in resistance again. Each one of these prominent features is suppressed when the magnetic field is applied, resulting in a smooth curve. After the curve in that region, the resistance continues to decrease as the temperature goes down. This is 5 Kelvin less than the non-magnetic field run prior, and can be considered to have been shifted and suppressed. On the final run, the magnetic field was removed, and we see that the drop in resistance most likely occurred at a higher temperature then our system achieves during runs (see N Low, Peel 003, Run 003 – MF and Run 004 in Appendix 13). At 319 Kelvin, the drop of resistance stops and increases until 306 Kelvin. From there it decreases to 280 Kelvin at which point, it increases to 230 Kelvin where it then starts to decrease until the bottom temperature. Comparing run 002 and run 004, we see that they have very similar features and Run 004 has a slightly lower resistance which is overlapping with the magnetic field run. This shows a residual effect of the magnetic field which causes it to maintain a lower resistance. This can partly be explained by pancake vortices melting. The presence of a magnetic field causes a suppression of features which returned when the magnetic field is removed. However, a change in the current flow is induced by the magnetic field, seen in following runs.

The high dose implantation with a concentration in HOPG of 5.71E+16 /cm³, did not have any signs of superconductivity (see N High in Appendix 14). There was a mild negative temperature coefficient but did not have any prominent steps or features. Lower doses of nitrogen ion implanted into HOPG warrants future investigation.

Beryllium is stripped of its 2 outermost electrons in the S orbital, prior to ion implantation, to give it a crystal ionic radius of 59 pm. On impact with the HOPG surface, the charge neutralization process returns the electrons, giving it an atomic radius of 112 pm. At this radius, with an energy of 10 keV, the projected range is 442 Å with a longitudinal straggle of 171 Å and lateral straggle of 130 Å. When this bivalent atom comes to rest, it becomes an electron acceptor for neighboring carbon atoms since it has 2 less valence electron.

For the beryllium low-dose sample with a concentration in HOPG of 2.81E+13 /cm³, there are no signs of superconductivity present (see Be Low in Appendix 15). The curve is featureless with a positive temperature coefficient. For the high dosage with concentration in HOPG of 2.81E+16 /cm³, the *R* vs. *T* response changed slightly, giving it a slightly negative temperature coefficient but still does not have any prominent features (see Be High in Appendix 16).

Fluorine is stripped of its 7 outermost electrons in the S and P orbitals, prior to ion implantation, to give it a crystal ionic radius of 22 pm. On impact with the HOPG surface, the charge neutralization process returns the electrons, giving it an atomic radius of 42 pm. At this radius, with an energy of 10 keV, the projected range is 193 Å with a longitudinal straggle of 68 Å and lateral straggle of 50 Å. When this univalent atom comes to rest, it has 3 more electrons than its neighboring carbon atoms but will accept an additional electron to fill its last orbital.

Fluorine low dosage with a concentration in HOPG of 7.06E+13 /cm³, has a very slight negative temperature coefficient that is featureless with no signs of superconductivity (see F Low in Appendix 17). The high dosage with concentration in HOPG of 7.06E+16 /cm³, has a positive temperature coefficient but it is also featureless as well as having no signs of superconductivity (see F High in Appendix 18).

Lithium is stripped of its outermost electron in the S orbital, prior to ion implantation, to give it a crystal ionic radius of 90 pm. On impact with the HOPG surface, the charge neutralization process returns the electrons, giving it an atomic radius of 167 pm. At this radius, with an energy of 10 keV, the projected range is 612 Å with a longitudinal straggle of 234 Å and lateral straggle of 187 Å. This is the furthest range for species that we have tested. When this univalent atom comes to rest, it becomes an electron acceptor for neighboring carbon atoms since it has 3 less valence electron.

Lithium low dosage with a concentration in HOPG of 2.05E+13 /cm³, has a very slight negative temperature coefficient that is featureless with no signs of superconductivity (see Li Low in Appendix 19). The high dosage with a concentration in HOPG of 2.05E+16 /cm³, has a positive temperature coefficient but it is featureless with no signs of superconductivity (see Li High in Appendix 20).

Sodium is stripped of its outermost electron in the S orbital, prior to ion implantation, to give it a crystal ionic radius of 116 pm. On impact with the HOPG surface, the charge neutralization process returns the electrons, giving it an atomic radius of 190 pm. At this radius, with an energy of 10 keV, the projected range is 169 Å with a longitudinal straggle of 58 Å and lateral straggle of 43 Å. When this univalent atom

comes to rest, it becomes an electron acceptor for neighboring carbon atoms since it has 3 less valence electron.

The low dosage of sodium ion implanted with a concentration in HOPG of 8.28E+13 /cm³, has the most extreme negative temperature coefficient out of the samples we have seen (see Na Low in Appendix 21). At our highest temperature of 345 Kelvin, the resistance is 1.8 milliohms. The resistance continues to go up, as temperature goes down until 58 Kelvin where are the resistance is 3.1 milliohms. It then goes down to a final resistance of 2.7 milliohms at 3 Kelvin which is still a higher resistance then the highest temperature. This may have potential as a semiconductor or in other applications where such a negative temperature coefficient is desirable.

For the high sodium dosage with a concentration in HOPG of 8.28E+16 /cm³, we see a small step at 235 Kelvin (see Na High in Appendix 22). Dosages in this range or higher may merit future investigation.

Sulfur is stripped of its 4 outermost electrons in the S and P orbitals, prior to ion implantation, to give it a crystal ionic radius of 51 pm. On impact with the HOPG surface, the charge neutralization process returns the electrons, giving it an atomic radius of 88 pm. At this radius, with an energy of 10 keV, the projected range is 125 Å with a longitudinal straggle of 38 Å and lateral straggle of 30 Å. When this hexavalent atom comes to rest, it becomes an electron donor for neighboring carbon atoms since it has 2 extra valence electrons.

Both low dosages and high dosages of sulfur which have a concentration in HOPG of 1.26E+14 /cm³ and 1.26E+14 /cm³ respectively, have a featureless negative

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temperature coefficient in their R vs. T curves (see S Low in Appendix 23 and S High in Appendix 24). No signs of superconductivity are present.

A response consistent with the presence of magnetic field flux vortices in phosphorous implanted Highly Oriented Pyrolytic Graphite and in phosphorous-doped exfoliated multilayer graphene has been observed. The melting of stacks of pancake vortices of varying lengths can account for the repeated nature of the observed steps in the Resistance versus Temperature characteristics of the material. The lack of zero resistance at low temperatures is also consistent with pancake vortex behaviour in the flux-flow regime. Thus the observed features can be described using the pancake vortex phenomenon. The presence of magnetic vortices requires, and is direct evidence of, superconductivity. The small Meissner effect may simply mean that the volume fraction of material involved is quite small or that the penetration depth is significantly larger than the sample thickness.

The material that was subjected to post doping argon implantation (damage) showed a discontinuous step in resistance at a temperature of about 265 K. The initial findings from prior work [72], led to the conclusion that the ultimate critical temperature in this system under test was in excess of 100 K and, may have very likely been considerably higher if damage incurred during the doping process was minimized. Clearly this conclusion has not been voided and, based on the material with phosphine in the Plasma Chemical Vapor Deposition growth gas mixture, may well be valid for a transition at a temperature of greater than 300 K.

It was observed that in addition to the first multilayer of Phosphine doped graphene, there was also present a great amount of graphite layered material adhering to the desired layers. This extra material may have the effect of acting as a parallel conductor, shunting the graphene transport to some degree, however we are unable to ascertain what effect this additional material has on the transport measurements with our current experimental apparatus.

VII. CONCLUSION

By using several known superconductors, we were able to verify that our cryogenic system was working correctly. When testing the YBa₂Cu₃O_{7-X} Target, we used indium between the Surface and probes. Even though this is not typically used for our HOPG and graphene samples, this did not change the measurement process and still allowed us to confirm proper system functionality. Another deviation was that we used 1 milliamp since YBa₂Cu₃O_{7-X} is a low conducting ceramic until it becomes superconducting. This changed the sensitivity of our Delta measurements when compared to our HOPG and graphene samples but showed that our transition temperature was accurately at 88 Kelvin. The thickness of the sample (3mm) caused the top surface to cool slower than the bottom and led to a cooling transition temperature of 21 Kelvin. During cooling, which is 18 times faster than warming, this becomes more noticeable. For warming, this temperature gradient becomes negligible.

When testing the YBa₂Cu₃O_{7-x} Hairpin filter which was 1 millimeter thick, we saw that there was a cooling transition temperature of 42 Kelvin and warming transition temperature of 92 Kelvin. Since the same current was used as the YBa₂Cu₃O_{7-x} target, we can assume that heating caused by current is equal and the cooling transition temperature being closer to the warming transition temperature is solely due to the sample being thinner. The lower temperature gradient assured us that for thin graphene peels, this will be significantly lower.

When testing our element type II superconductor niobium, we found that our warming transition temperature was 9.244 Kelvin which is 0.056 Kelvin from its known transition temperature of 9.3 Kelvin. This further confirms that our thermocouples are working correctly and have high accuracy. We also saw that our system was capable of measuring an instantaneous drop in resistance even at very low temperatures. This one was also important for refining our set up to include a universal power supply which would continue measurements during power failures.

We learned that our Keithley Model 6221 will blink or source zero current with a blue indicator light on the front panel if there is an open circuit due to a loss of contact. The measurement will also drop to zero in voltage causing a erroneous reading in the resistance since there is no current running through the material. This happens virtually instantaneously due to thermal contraction. Although this was not desired, this shows us that small or gradual steps we were to see in future runs could not simply be explained by loss of contact.

For our low dose ion implantation of arsenic, we saw a change in slope at 255 Kelvin which continues to 148.77 Kelvin. There could be a very gradual transition occurring. With a magnetic field applied, the change in slope happened more gradually but did not create a change conclusive enough to determine if there is flux flow present. The higher dose of Arsenic ion implantation did not appear to have this effect. Exploring a lower dose of arsenic could potentially enhance the features we saw or simply

minimizing the damage that could be lowering the coherence length, by using a Chemical Vapor Deposition arsenic graphene growth.

On our aluminum ion implanted sample with low dosage, we see an abrupt step at 264 Kelvin which ends at 210 Kelvin. When the magnetic field was applied, this step was suppressed by 14 Kelvin. The end of the transition was suppressed by 9 Kelvin. This shows a clear change caused by the magnetic field. On the following run, when the magnetic field was removed, there appears to be residual effects that cause the run to look identical to the magnetic field run. This can be explained by residual vortices that were induced by the prior run. For the higher dosage of arsenic, we do not see this step. This is most likely due to more damage in the sample. For future work, a stronger magnetic field could be applied to see if the step gets suppressed further. A higher grade of HOPG (ZYA) could be explored as well as Chemical Vapor Deposition growth with aluminum present.

The low dose ion implantation of nitrogen showed a negative temperature coefficient with a step a 283 Kelvin and ending at 261 Kelvin. When the magnetic field is applied, we see that instead of an abrupt step it changes to a gradual slope near the transition apex. Outside of that, the 2 runs are very close to each other and even overlapping at higher temperatures. When the magnetic field was removed, the following run was identical to the previous magnetic field run. There appears to be a residual effect caused by the magnetic field. For future work, a stronger magnetic field should be applied since there was not a strong change when the magnetic field we used was present.

Upon further investigation, we did several peels to test the 2D characteristics that would closely approximate multi-layered grapheme [420]. There appeared to be 3 prominent changes in the slope that were linearly connected. This multi-step region occurred between 329 Kelvin and 235 Kelvin. These steps disappear when the magnetic field is applied, and just turns to a smooth curve in that region. The overall resistance also lowers when the magnetic field is applied in that region butt on the following run when the magnetic field is removed, the features return and the overall resistance in that region remains identical to what it was with the field applied. This shows that there was a residual effect that occurred from the magnetic field that affected the resistance but still allowed the features to return. The fact that the magnetic field change the response warrants future investigation. The higher dose appears to be featureless due to excess damage.

For the beryllium low dose bulk sample, there are no features present. For the high dosage, it changes to a negative temperature coefficient but does not show any features. There are no signs of superconductivity present.

Fluorine had a slight negative temperature coefficient at low dosages but a positive temperature coefficient at high dosages. Both were featureless and did not show signs of superconductivity.

Lithium had a slight negative temperature coefficient at low dosages but a positive temperature coefficient at high dosages. Both were featureless and did not show signs of superconductivity.

For the low dosage of sodium, we see an extremely negative temperature coefficient. Even after the freeze out region, the resistance was higher then the resistance at 345 Kelvin. There were no features present and there are no signs of superconductivity.

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For the high dosage of sodium, we saw a small step at 235 Kelvin. This could be a transition for just several layers in the bulk HOPG that is being shunted by non superconducting layers in the HOPG. Testing with a magnetic field and removing several layers while testing each layer should be done to explore this feature.

Both low dosages and high dosages of sulphur yielded a negative temperature coefficient but did not show any features. There are no signs of superconductivity present.

For the phosporous doped samples the repeated steps in the resistance versus temperature characteristics can be seen to be independent of the manner in which the phosphorous-doped HOPG and graphene were doped. These steps become discontinuous at elevated temperatures when Argon (damage) is added. This is consistent with the results that are expected for thin superconducting films with and without damage.

The observed electrical resistance and magnetic behaviours of the samples under test can be seen to be consistent with superconductivity, even though a lack of zero resistance at low temperature was observed. In fact, the high degree of anisotropy in graphite and graphene predicts that if the material is a superconductor there would be significant magnetic flux-flow losses down to very low temperatures [68], [69]-[71].

The magnetic response of phosphorous-doped HOPG and graphene is as would be expected for superconductors of similar physical characteristics. That is (1) that the superconducting region is thin in comparison to the London (magnetic) penetration depth and (2) that the high level of anisotropy in the material is favourable for the formation of pancake vortices. The observed quenching of the R vs. T curve by application of a magnetic field is also to be expected for a superconductor.

The magnetization and susceptibility results for phosphorous-doped HOPG and graphene suggests (1) a transition temperature above 260 K for the magnetization based upon the Zero-Field Cooled to Field Cooled Hysteresis loop and (2) a transition temperature above 150 K based upon the AC susceptometer measurements. There is no trace of ferromagnetism in the results as both the magnetization and susceptibility are negative in the lower temperature region.

In going from room temperature to a temperature of 80K, The Hall voltage has been observed to undergo a sign change during Hall Effect measurements. This phenomenon has been observed in other, known and accepted, superconductors that are in the flux-flow region of the mixed or vortex state [356]-[386]. It is also seen in anomalous ferromagnetism. Since there are (1) no ferromagnetic atoms and (2) no atoms with d or f electron shells in the sample, it is highly unlikely that ferromagnetism is involved in these Hall measurements. The fact that the magnetization and magnetic susceptibility are both negative suggests that ferromagnetism played no part in the sign reversal in the Hall voltage.

The aforementioned evidence forces us to conclude that phosphorous-doped Highly Oriented Pyrolytic Graphite (and phosphorous-doped graphene) is a superconductor with a transition temperature between 150 K and 260 K. Some of the data acquired hints at a possibility that the transition temperature in the best samples may approach room temperature.

In summary, we were the first to systematically and exhaustively dope graphene for the purposes of creating a high temperature superconductor. We were the first to study the characteristics of Plasma Enhanced Chemical Vapor Deposited graphene in a phosphine environment due to our custom system built and certified for that purpose. We successfully built a cryogenic system that can achieve 2.5 Kelvin measurements, all the way up to 348 Kelvin, with a removable magnetic field coil system that does not disturb the sample. Detailed software was created from scratch to automate almost every aspect of our delta measurements to ensure consistency while also providing convenience. This software successfully controlled voltage measurements, current generation, compressor functionality and monitoring, temperature monitoring and heaters to allow above room temperature measurements. We were not successful in creating a high accuracy susceptometer due to ambient noise, however, we compensated by sending our samples to an external research facility to achieve the same goals.

- We have created the highest transition temperature on record, of upwards of 260 Kelvin with indications of near room temperature transitions using perfected doping techniques.
- All of the pentavalent electron donors showed signs of superconductivity (phosphorus, nitrogen, arsenic). These showed strongest features for low dosage.
- We saw that flux vortices were created in doped graphene, indicating it is a type II superconductor.
- As expected, Plasma Enhanced Chemical Vapor Deposition doping minimized the damage to our graphene lattice and allowed longer coherence lengths than ion implanted doping. This also gave us flux flow which added to our resistance and prevented us from seeing a zero resistance superconductor.
- Ion Implanted doping created columnar defects that pinned our flux vortices. This created pancake vortices that melted off in stages, causing multiple steps.

• The mixed state region appeared to be much larger than we expected, with high temperature boundaries upwards of 260 Kelvin and low temperature boundaries unmeasurable by our equipment (may occur at temperatures under 3 Kelvin or not at all). This is the first time this phenomenon has been seen.

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APPENDICES

Appendix 1 *R* vs. *T* for YBa₂Cu₃O_{7-X} Target



Appendix 2 *R* vs. *T* for YBa₂Cu₃O_{7-X} Hairpin



Appendix 3 *R* vs. *T* for Niobium



Appendix 4 Loss of Contact


Appendix 5 As Low Peel



Appendix 6 As Hi Peel



Appendix 7 Al Low Bulk Run 002 and Run 003 - MF



Appendix 8 Al Low Bulk Run 003-MF and Run 004



Appendix 9 Al High



Appendix 10 N Low, Peel 000, Run 002 and Run 003 – MF



Appendix 11 N Low, Peel 000, Run 003 – MF and Run 004



Appendix 12 N Low, Peel 003, Run 002 and Run 003 – MF



Appendix 13 N Low, Peel 003, Run 003 – MF and Run 004



Appendix 14 N High



Appendix 15 Be Low



Appendix 16 Be High



Appendix 17 F Low



Appendix 18 F High



Appendix 19 Li Low



Appendix 20 Li High



Appendix 21 Na Low



Appendix 22 Na High


Appendix 23 S Low



Appendix 24 S High



Appendix 25 Flow Chart for Cryo4-Delta Measure ver 23 Auto compressor

and two separate files generated- VI name was disconnected on cooling in

previous version.vi





Appendix 26 Flow Chart For - Header Gen (SubVI) version 2 - run logger

attempt to add comments.vi



Appendix 27 Flow Chart For - R vs T SubVI LakeShore part - SubVI for R

vs T run v3 - Case structure removed.vi



Appendix 28 Flow Chart For - RunCalculatorINI.vi



Appendix 29 Cryo3- Magnetometer Ver 2.vi



Cryo3- Magnetometer Ver 2-1.vi







Inserts a time delay into the calling VI.

This Express VI is configured as follows:

Delay Time: 30 s



Build XY Graph

Build XY Graph formats the data displayed on an X-Y Graph.







Convert from Dynamic Data

Convert from Dynamic Data Converts the dynamic data type to numeric, Boolean, waveform, and array data types for use with other VIs and functions.

1	₹	
d,	9	n.

Wait Wait

Delays for a certain time interval before the output data dependence becomes valid.



Wait2 Wait

Delays for a certain time interval before the output data dependence becomes valid.

Appendix 30 Cry03- SubVI: SR7270 8-bit R232 comms.vi



SR7270 8-bit RS232 comms.vi C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\SR7270LV8.llb\SR7270 8-bit RS232 comms.vi Last modified on 5/11/2009 at 7:01 PM Printed on 5/20/2016 at 8:01 PM

Connector Pane

SR7270 8-bit RS232 comms.vi

VISA session (for class)	7270	VISA session
error in (no error)	38400 RS232	error out

This VI forces the RS232 settings in the instrument and current VISA session, if this is using RS232, to 38400 baud ar 8 data bits, allowing subsequent binary dump commands, that respond with 8-bit data, to work correctly.

12 May 2009: First release

Front Panel

VISA session (for class)	Set RS232 Parameters to 8-bit, 34000 Baud	VISA session
error in (no error) status code no error 0 source	error out status no error source	code 0



SR7270 8-bit RS232 comms.vi C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\SR7270LV8.llb\SR7270 8-bit RS232 comms.vi Last modified on 5/11/2009 at 7:01 PM Printed on 5/20/2016 at 8:01 PM

Block Diagram



Appendix 31 Cryo3- SubVI: SR7270 Configure Reference Channel-Single

Reference.vi

Page 1 7270 ref chan

SR7270 Configure Reference Channel - Single Reference.vi C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\SR7270LV8.llb\SR7270 Configure Reference Channel - Single Reference.vi Last modified on 5/11/2009 at 6:58 PM Printed on 5/20/2016 at 8:02 PM

Connector Pane

SR7270 Configure Reference Channel - Single Reference.vi



This VI controls the reference channel signal source, oscillator amplitude and frequency, reference phase and harmonic for either a single reference channel or for channel 1 when using the dual reference or dual harmonic modes

12 March 2009: First Release

Page 2 7270 ref chan

SR7270 Configure Reference Channel - Single Reference.vi C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\SR7270LV8.llb\SR7270 Configure Reference Channel - Single Reference.vi Last modified on 5/11/2009 at 6:58 PM Printed on 5/20/2016 at 8:02 PM

Front Panel



Page 3 7270 ref chan

SR7270 Configure Reference Channel - Single Reference.vi C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\SR7270LV8.llb\SR7270 Configure Reference Channel - Single Reference.vi Last modified on 5/11/2009 at 6:58 PM Printed on 5/20/2016 at 8:02 PM



Page 4 7270 ref chan

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Channel - Single Reference.vi

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Printed on 5/20/2016 at 8:02 PM 7 [0..8] -V Į Checks Reference Phase if AQN has run of if update boolean (set by Auto-measure) is set)v 🖣 True 🗕 REFP1 沙 REFP 1000 7270 VISA C





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Appendix 32 Cryo3- SubVI: SR7270 Configure Sensitive-Single Reference.vi
Page 1 7270 set sen

SR7270 Configure Sensitivity - Single Reference.vi C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\SR7270LV8.IIb\SR7270 Configure Sensitivity -Single Reference.vi Last modified on 5/11/2009 at 6:58 PM Printed on 5/20/2016 at 8:03 PM

Connector Pane

SR7270 Configure Sensitivity - Single Reference.vi



This control sets the full-scale sensitivity of the signal channel, or that of the first channel in the dual reference or harmonic modes. It also sets the AC gain

12 March 2009: First Release

Page 2 7270 set sen

SR7270 Configure Sensitivity - Single Reference.vi C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\SR7270LV8.llb\SR7270 Configure Sensitivity -Single Reference.vi Last modified on 5/11/2009 at 6:58 PM Printed on 5/20/2016 at 8:03 PM

Front Panel



Page 3 7270 set sen

SR7270 Configure Sensitivity - Single Reference.vi C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\SR7270LV8.llb\SR7270 Configure Sensitivity -Single Reference.vi Last modified on 5/11/2009 at 6:58 PM Printed on 5/20/2016 at 8:03 PM

Block Diagram



Page 4 7270 set sen

SR7270 Configure Sensitivity - Single Reference.vi

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SR7270 Configure Sensitivity - Single Reference.vi

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SR7270 Configure Sensitivity - Single Reference.vi

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SR7270 Configure Sensitivity - Single Reference.vi

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SR7270 Configure Sensitivity - Single Reference.vi

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Single Reference.vi Last modified on 5/11/2009 at 6:58 PM

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	Stores present settings so that if routine is repeatedly called unnecessary commands are not sent	F
		ē
D		
▶ † Prev	ious Range	
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▶ ↑ Prev	ious AC Gain	
• 		
► † Previ	ous Auto AC Gain	
h .		

Appendix 33 Cryo3- SubVI: SR7270 Configure Signal Channel Input.vi

Page 1 7270 signal chan

SR7270 Configure Signal Channel Input.vi C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\SR7270LV8.IIb\SR7270 Configure Signal Channel Input.vi Last modified on 5/11/2009 at 6:57 PM Printed on 5/20/2016 at 8:04 PM

Connector Pane

SR7270 Configure Signal Channel Input.vi



This VI configues the input signal channel and the line-frequency rejection filter.

12 March 2009: First Release

Front Panel

VISA session (for class)	Signal Channel - Input Configuratio Update Line Frequency Rejection Filter	n VISA session
A (Single-Ended Voltage)	OFF	0
Input Connector Shells Float Ground Input Device FET Bipolar	Line Frequency 50 Hz 60 Hz	
error in (no error)	err	or out
status code	s: Q	tatus code gerror ₀
source	so	urce

Page 2 7270 signal chan

SR7270 Configure Signal Channel Input.vi C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\SR7270LV8.llb\SR7270 Configure Signal Channel Input.vi Last modified on 5/11/2009 at 6:57 PM Printed on 5/20/2016 at 8:04 PM

Block Diagram



SR7270 Configure Signal Channel Input.vi C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\SR7270LV8.IIb\SR7270 Configure Signal Channel Input.vi Last modified on 5/11/2009 at 6:57 PM Page 3 7270 signal chan



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Page 5 7270 signal chan

SR7270 Configure Signal Channel Input.vi

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Page 6 7270 signal chan

SR7270 Configure Signal Channel Input.vi

C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\SR7270LV8.llb\SR7270 Configure Signal Channel Input.vi



Page 7 7270 signal chan

SR7270 Configure Signal Channel Input.vi

C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\SR7270LV8.llb\SR7270 Configure Signal Channel Input.vi

▶ ♠ Previous Input Mode Stores present settings so that if routine is repeatedly called unnecessary commands are not sent
Previous Input Connector Shells
► Previous Line Frequency Rejection Filter
► Previous Line Frequency
► Previous Input Device

Appendix 34 Cryo3- SubVI: SR7270 Configure Virtual Reference Mode.vi

SR7270 Configure Virtual Reference Mode.vi C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\SR7270LV8.llb\SR7270 Configure Virtual Reference Mode.vi Last modified on 5/11/2009 at 7:05 PM Printed on 5/20/2016 at 6:39 PM



Connector Pane

SR7270 Configure Virtual Reference Mode.vi

VISA session (for class) 🚽	7270	VISA session
Virtual Reference Mode $igsqcup$	v ref	
error in (no error) 🚅	mode	error out

This VI turns the Virtual Reference Mode on or off.

12 March 2009: First Release

Front Panel

VISA session (for class)	VISA session
	Virtual Reference Mode On
error in (no error)	Off error out
status code	status code no errov 0
source	source

Page 2 7270 v ref mode

SR7270 Configure Virtual Reference Mode.vi C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\SR7270LV8.IIb\SR7270 Configure Virtual Reference Mode.vi Last modified on 5/11/2009 at 7:05 PM Printed on 5/20/2016 at 6:39 PM

Block Diagram



Page 3 7270 v ref mode

SR7270 Configure Virtual Reference Mode.vi C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\SR7270LV8.llb\SR7270 Configure Virtual Reference Mode.vi Last modified on 5/11/2009 at 7:05 PM Printed on 5/20/2016 at 6:39 PM

200000000000000000000000000000000000000		
1	Stores present settings so that if routine is	
Г	repeatedly called unnecessary commands are	
	not sent	
► Previous Virtual Refe	rence Mode	
1		
3		6
8 1		
Г		-

Appendix 35 Cryo3- SubVI: SR7270 Initialization.vi



SR7270 Initialize.vi C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\SR7270LV8.llb\SR7270 Initialize.vi Last modified on 5/11/2009 at 6:57 PM Printed on 5/20/2016 at 8:05 PM

Connector Pane

SR7270 Initialize.vi



This VI passes the addressing information in the instrument decriptor to the Instr Open VI and returns the instrument ID. You can optionally reset the instrument by setting the front panel reset control. You must run this VI before usi any of the instrument driver VIs for this instrument.

12 May 2009: First Release







SR7270 Initialize.vi C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\SR7270LV8.llb\SR7270 Initialize.vi Last modified on 5/11/2009 at 6:57 PM Printed on 5/20/2016 at 8:05 PM

Block Diagram



Appendix 36 Cryo3- SubVI: SR7270 VISA WriteRead.vi



Connector Pane

SR7270 VISA Write/Read.vi



This VI should be used instead of standard VISA write or write-read constructs since it supports the software handshaking required when using the RS232 interface. It also handles correctly the USB and Ethernet interfaces, and the following types of instrument response:-Single response, e.g. to X (output) command Multiple responses, e.g. to M (monitor) command Array responses, e.g. to DC (dump curve) command Response to * (speed transfer) command

It also reports the instrument's status

12 May 2009: First Release



Front Panel

	VISA session (for class) Translate DC. n to Write Command String Write Command String	SEN; DCB n Output Data Array	VISA session
	Invalid Command Command Parameter error Reference Unlock Input Overload Output Overload ADC Triggered	Response 1 Response 2 Response 3 Response 4	error out status code no errog 0 source
error in (no error)			
source			



Block Diagram







Page 5 7270 VISA @ Wrt/rd



Page 6 7270 VISA 6 Wrt/rd



Page 7 7270 VISA C Wrt/rd



Page 8 7270 VISA 6 Wrt/rd



Page 9 7270 VISA (S Wrt/rd



Page 10 VISA & Wrt/rd




SR7270 VISA Write/Read.vi C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\SR7270LV8.IIb\SR7270 VISA Write/Read.vi Last modified on 5/11/2009 at 7:02 PM Printed on 5/20/2016 at 8:00 PM



Appendix 37 Cryo4-Delta Measure ver 23 Auto compressor and two

separate files generated- VI name was disconnected on cooling in previous

version.vi



Cryo4-Delta Measure ver 23 Auto compressor and two separate files generated- VI name was disconnecte C:\Users\LabVIEW-user\AppData\Local\Temp\Cryo4-Delta Measure ver 23 Auto compressor and two separate files generated- VI name was disconnected on cooling in previous version.vi Last modified on 5/20/2016 at 5:21 PM Printed on 5/20/2016 at 5:36 PM

Cryo4-Delta Measure ver 23 Auto compressor and two separate files generated- VI name was disconnected





Cryo4-Delta Measure ver 23 Auto compressor and two separate files generated- VI name was disconnecte C:\Users\LabVIEW-user\AppData\Local\Temp\Cryo4-Delta Measure ver 23 Auto compressor and two separate files generated- VI name was disconnected on cooling in previous version.vi Last modified on 5/20/2016 at 5:21 PM Printed on 5/20/2016 at 5:36 PM

on cooling in previous version.vi



Cryo4-Delta Measure ver 23 Auto compressor and two separate files generated- VI name was disconnecte C:\Users\LabVIEW-user\AppData\Local\Temp\Cryo4-Delta Measure ver 23 Auto compressor and two separate files generated- VI name was disconnected on cooling in previous version.vi Last modified on 5/20/2016 at 5:21 PM Printed on 5/20/2016 at 5:36 PM



Appendix 38 F70 read all and command_SubVI.vi



Connector Pane

F70 read all and command_SubVI.vi



F-70 compressor RS232 communication driver.

This VI can directly output reading of T1, T2, T3, P1, P2, Firmware version, operation hours, and compressor statu: (state of operation, alarms etc.). It can also send operation command (turn ON/OFF compressor and cold head, et when compressor is in configuration mode 1.

It can be used as a sub-vi in other program.

It takes two inputs:

1. VISA resource name (COM1, COM2, etc.)

2. F70 command (you can choose from the drop down list (Turn on compressor and cold head, turn of compressc and cold head, cold head pause, read all temperatures, read all pressures, read status bits etc.)

It outputs the following (depending on the input F70 command):

- 1. Compressor temperature (T1, T2, T3)
- 2. Compressor Pressure (P1, P2)
- 3. Compressor status bits (configuration mode, state of operation, alarms, system on, etc.)
- 4. Firmware version and operation hours.

General Information about F-70 RS232 communication protocol.

This section describes the F-70 compressor RS232 interface only. It does not describe overall operation or safety of the F-70 compressor. Please refer to the F-70 operating manual for operating instructions and warnings. The information in this section should be used only after safe operation of the F-70 compressor is understood.

1. F-70 Front Panel Connection: Male DB-9 connector Pin 2 = Receive Pin 3 = Transmit Pin 5 = Earth Ground (connected to compressor chassis ground)

2. RS232 parameters. a. Baud - 9600 b. No parity c. 8 bit data

d. 1 bit stop



The F-70 sends no unsolicited messages. It only responds to commands from a host computer.

NOTE

RS232 commands that change operation of the F-70 compressor (on, off, reset, cold head run, cold head pause a pause off) have equal value as front panel key press control. The F-70 will respond equally to either source of contr input in the order in which it is received.

NOTE

There are no error routines or checks to prevent possible conflicts if a compressor is both connected to a diagnostic interface (DB25) control element and an RS232 control element except:

1. RS232 commands that change operation of the F-70 compressor are active only when the compressor is set for DB25 Configuration Mode 1 (reference operating manual). RS232 commands that transmit data are active for bo DB25 Configuration Mode 1 and Mode 2.

2. In Configuration Mode 1, active DB25 operating signals will take priority over front panel key press and RS232 control. DB25 Configuration Mode 2 does not permit either RS232 or front panel key press control.

RS232 Command List (See the Command Set section below for command descriptions and examples)

Information Commands
 \$TEA: Read all temperatures \$TEn: Read temperature n (n = 1, 2, 3, or 4)
 \$PRA: Read all pressures \$PRn: Read pressure n (n = 1 or 2)
 \$STA: Read status bits \$ID1: Read firmware version and elapsed operating hours

2. Operating Commands
\$ON1: On \$OFF: Off
\$RS1: Reset \$CHR: Cold head run
\$CHP: Cold head pause \$POF: Cold head pause off

3. Responses: \$???: Invalid command received

Command Structure

The individual bytes of any communication packet (frame) are restricted to the ASCII domain of 0x20 (20H, space through 0x7E (7EH, tilde), plus 0x0D (carriage return).

The basic communication frame from the host computer to the F-70 compressor is comprised of a start character command mnemonic, data (if required), checksum, and a message end character.

Start Character: This is always 0x24 (24H, dollar sign).

Command: This is a 3 character mnemonic. It defines the action which will be taken by the controller.

Data: Data will be transmitted as ASCII text equivalents. Example: the number 123 will be text "123". In the event t



a floating point number is to be conveyed, then it will also be in plain text. Example: 123.9 will be text "123.9". The data field length is fixed depending on the command that is issued. This will be defined at the command level.

Checksum (CRC-16): The checksum is a 16 bit CRC (CRC-16). It is transmitted as four-digit ASCII hex. Example: a bit binary checksum "001001110101011" in four-digit ASCII equals "23AB". The check sum calculation includes the "\$" start character, command field, and data field (if present). See section E below for description of CRC-16 generation.

End of message: This is always 0x0D (0DH carriage return). This assures that the end of message character falls outs the standard text domain and will not be incorporated in commands, data, or checksums.

The response frame from the F-70 compressor is similar to the command frame, but includes delimiters between various fields.

Start Character: This is always 0x24 (24H, dollar sign).

Command: This is a 3 character mnemonic. It is the same as (echoes) the command sent from the host controller

Delimiter: This is always 0x2C (2CH, comma).

Data: Data will be transmitted as ASCII text equivalents. Example: the number 123 will be text "123". In the event t a floating point number is to be conveyed, then it will also be in plain text. Example: 123.9 will be text "123.9". The data field length is fixed depending on the command that is issued. This will be defined at the command level.

Checksum(CRC-16): The checksum is a 16 bit CRC. It is transmitted as four-digit ASCII hex. Example: a 16 bit bin checksum "001001110101011" in four-digit ASCII equals "23AB". The check sum calculation includes the "\$" start character, command field, data fields (if present), and all commas including the comma preceding the checksum. See section E below for description of CRC-16 generation.

End of message: This is always 0x0D (0DH carriage return). This assures that the end of message character falls outs the standard text domain and will not be incorporated in commands, data, or checksums.

Command Set

\$TEA: Read all temperatures Command with checksum and carriage return = \$TEAA4B9<cr> Response: \$TEA,T1,T2,T3,T4,<crc-16><cr>

T1 through T4 are compressor internal temperatures in degrees C.

T1 = Compressor capsule helium discharge temperature

T2 = Water outlet temperature

T3 = Water inlet temperature

T4 is inactive (returns "000") for most F-70 variants.



T1 through T4 are compressor internal temperatures in degrees C.

- T1 = Compressor capsule helium discharge temperature
- T2 = Water outlet temperature
- T3 = Water inlet temperature

T4 is inactive (returns "000") for most F-70 variants.

The temperature fields are always 3 characters long and are rounded to the nearest degree. Temperatures less that 100 °C have leading zeroes.

Example: TEA,086,040,031,000,3798 < cr> corresponding to T1 = 86°C, T2 = 40°C, T3 = 31°C and where 3798 is the checksum and <cr> is the carriage return.

\$Ten: Read selected temperature (n = 1, 2, 3, or 4)
Command with checksum and carriage return = \$TE140B8<cr>, \$TE241F8<cr>, \$TE38139<cr>, or \$TE44378<cr>
Response: \$TEn,Tn,<crc-16><cr>

Example: host sends TE140B8cr. The response from the compressor is TE1,086,ADBC < cr> corresponding to temperature T1 = 86°C and where ADBC is the checksum and <math>< cr> is the carriage return.

\$PRA: Read all pressures

Command with checksum and carriage return = \$PRA95F7<cr> Response: \$PRA,P1,P2,<crc-16><cr>

P1 is the compressor return pressure in PSIG. P2 is inactive (returns "000") for most F-70 variants.

The pressure fields are always 3 characters long and are rounded to the nearest whole number. Pressures less than 100 psig have leading zeroes.

Example: \$PRA,079,000,0CEC < cr> corresponding to P1 = 79 psig and where 0CEC is the checksum and < cr> is the carriage return.

\$PRn: Read selected pressure (n = 1 or 2)
Command with checksum and carriage return = \$PR171F6<cr> or \$PR270B6<cr>
Response: \$PRn,Pn,<crc-16><cr>

Example: host sends PR171F6 < cr>. The response from the compressor is PR1,079,2EBD < cr> corresponding to pressure P1 = 79 psig and where 2EBD is the checksum and < cr> is the carriage return.

\$STA: Read Status bits Command with checksum and carriage return = \$STA3504<cr>

Response: \$STA, status bits, <crc-16> <cr>



T1 through T4 are compressor internal temperatures in degrees C.

- T1 = Compressor capsule helium discharge temperature
- T2 = Water outlet temperature
- T3 = Water inlet temperature

T4 is inactive (returns "000") for most F-70 variants.

The temperature fields are always 3 characters long and are rounded to the nearest degree. Temperatures less that 100 °C have leading zeroes.

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Response: \$STA, status bits, <crc-16> <cr>



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- T1 = Compressor capsule helium discharge temperature
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- T3 = Water inlet temperature

T4 is inactive (returns "000") for most F-70 variants.

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Example: host sends PR171F6 < cr>. The response from the compressor is PR1,079,2EBD < cr> corresponding to pressure P1 = 79 psig and where 2EBD is the checksum and < cr> is the carriage return.

\$STA: Read Status bits Command with checksum and carriage return = \$STA3504<cr>

Response: \$STA, status bits, <crc-16> <cr>



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- T1 = Compressor capsule helium discharge temperature
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Command with checksum and carriage return = \$TE140B8<cr>, \$TE241F8<cr>, \$TE38139<cr>, or \$TE44378<cr>
Response: \$TEn,Tn,<crc-16><cr>

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Example: host sends PR171F6 < cr>. The response from the compressor is PR1,079,2EBD < cr> corresponding to pressure P1 = 79 psig and where 2EBD is the checksum and < cr> is the carriage return.

\$STA: Read Status bits Command with checksum and carriage return = \$STA3504<cr>

Response: \$STA, status bits, <crc-16> <cr>



T1 through T4 are compressor internal temperatures in degrees C.

- T1 = Compressor capsule helium discharge temperature
- T2 = Water outlet temperature
- T3 = Water inlet temperature

T4 is inactive (returns "000") for most F-70 variants.

The temperature fields are always 3 characters long and are rounded to the nearest degree. Temperatures less that 100 °C have leading zeroes.

Example: TEA,086,040,031,000,3798 < cr> corresponding to $T1 = 86^{\circ}C$, $T2 = 40^{\circ}C$, $T3 = 31^{\circ}C$ and where 3798 is the checksum and <cr> is the carriage return.

\$Ten: Read selected temperature (n = 1, 2, 3, or 4)
Command with checksum and carriage return = \$TE140B8<cr>, \$TE241F8<cr>, \$TE38139<cr>, or \$TE44378<cr>
Response: \$TEn,Tn,<crc-16><cr>

Example: host sends TE140B8cr. The response from the compressor is TE1,086,ADBC < cr> corresponding to temperature T1 = 86°C and where ADBC is the checksum and <math>< cr> is the carriage return.

\$PRA: Read all pressures

Command with checksum and carriage return = \$PRA95F7<cr> Response: \$PRA,P1,P2,<crc-16><cr>

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Example: \$PRA,079,000,0CEC < cr> corresponding to P1 = 79 psig and where 0CEC is the checksum and < cr> is the carriage return.

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Response: \$PRn,Pn,<crc-16><cr>

Example: host sends PR171F6 < cr >. The response from the compressor is PR1,079,2EBD < cr > corresponding to pressure P1 = 79 psig and where 2EBD is the checksum and < cr > is the carriage return.



Front Panel



Page 10 F 70 COM ALL







H (Q C 1 and FL look (Folum) 12 (p.)



Block Diagram







)	¥i	-+	era Dr
		-H	
)			
	H .	Cold Head Run	
)	¥!	Cold Head Fease	
)	<u>H</u> (-#	Fact Crt
)		-H Osfestor	

Appendix 39 Fast Sweep -Kiar v17 changed from Pseudo to differential.vi

Page 1 FAST DAQMX SWEEP

Fast Sweep -Kiar v17 changed from Pseudo to differential-2.vi C:\Users\LabVIEW-user\AppData\Local\Temp\Fast Sweep -Kiar v17 changed from Pseudo to differential-2.vi Last modified on 5/20/2016 at 6:05 PM Printed on 5/20/2016 at 6:06 PM

Connector Pane

Fast Sweep -Kiar v17 changed from Pseudo to differential-2.vi



Page 2 FAST DAQMX SWEEP

Fast Sweep -Kiar v17 changed from Pseudo to differential-2.vi C:\Users\LabVIEW-user\AppData\Local\Temp\Fast Sweep -Kiar v17 changed from Pseudo to differential-2.vi Last modified on 5/20/2016 at 6:05 PM

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I. Description:

This example demonstrates how to continuously output a waveform using an internal sample clock. The automati regeneration of data has been disabled, so new data has to be provided throughout the duration of the continuous Analog Output operation. This allows the generation of any arbitrary frequency as this VI computes new data for each iteration of the loop, maintaining phase continuity of the signal.

II. Instructions for Running:

1. Select the Physical Channel to correspond to where your signal is output on the DAQ device.

2. Enter the Minimum and Maximum Voltage range.

3. Specify the desired Sample Clock Rate of the output Waveform. Higher sample clock rates will produce a smoother waveform.

4. Manually specify the Analog Output buffer size, in number of samples. A larger buffer would help non-regenerative operations avoid errors due to occasional high CPU load.

Note:

The Analog Output buffer has to be at least as big as the amount of data being written to the board per iteration. T recommended buffer size would be at least twice the amount of data being written to the board per iteration. 5. Enter the Waveform Information. See the documentation of the Basic Function Generator SubVI for informatio

on each parameter.

III. Block Diagram Steps:

1. Create an Analog Output Voltage channel.

2. Configure the task to prohibit the automatic regeneration of data.

3. Call the DAQmx (Sample Clock) VI to set the sample clock rate. Additionally, set the sample mode to Continuou 4. Read the actual sample clock rate (eventually coerced depending on the hardware used).

5. Compute the desired waveform, using the buffer size and the actual update rate. This VI keeps track of the phase the waveform to ensure that the generated signal is continuous.

6. Write the waveform to the output buffer.

7a. Call the Start VI. This is only needed when the loop is executed for the first time.

7b. Do nothing.

8. Loop continuously until user presses the Stop button. Every iteration computes and writes a new waveform to t buffer.

9. Call the Clear Task VI to clear the Task.

10. Use the popup dialog box to display an error or warning if any.

IV. I/O Connections Overview:

Make sure your signal output terminal matches the Physical Channel I/O Control. For further connection information, refer to your hardware reference manual.

Page 3 FAST DAGMX SWEEP

Fast Sweep -Kiar v17 changed from Pseudo to differential-2.vi C:\Users\LabVIEW-user\AppData\Local\Temp\Fast Sweep -Kiar v17 changed from Pseudo to differential-2.vi Last modified on 5/20/2016 at 6:05 PM Printed on 5/20/2016 at 6:06 PM

Front Panel



Page 4 FAST DAQMX SWEEP

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Page 5 FAST DAQMX SWEEP

Fast Sweep -Kiar v17 changed from Pseudo to differential-2.vi C:\Users\LabVIEW-user\AppData\Local\Temp\Fast Sweep -Kiar v17 changed from Pseudo to differential-2.vi Last modified on 5/20/2016 at 6:05 PM Printed on 5/20/2016 at 6:06 PM

Block Diagram





Appendix 40 Fast Sweep v7 Current step and resistor added to csv.vi



Fast Sweep v7 Current step and resistor added to csv.vi



Calculates the current from the total number of steps.









Convert to Dynamic Data

Convert to Dynamic Data

Converts numeric, Boolean, waveform and array data types to the dynamic data type for use with Express VIs.







[46]⁽Convert to Dynamic Data2

Convert to Dynamic Data Converts numeric, Boolean, waveform and array data types to the dynamic data type for use with Express VIs.

Appendix 41 Find Start and End (SubVI).vi

Page 1 FIND START

Find Start and End (SubVI).vi C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\Find Start and End (SubVI).vi Last modified on 5/20/2016 at 6:05 PM Printed on 5/20/2016 at 6:15 PM

Find Start and End (SubVI).vi





Page 2

Find Start and End (SubVI).vi C:\Users\LabVIEW-user\Desktop\KELVIN & TUA\Kelvin\Find Start and End (SubVI).vi Last modified on 5/20/2016 at 6:05 PM Printed on 5/20/2016 at 6:15 PM



Appendix 42 GmailAttachmentSender.vi


GmailAttachmentSender.vi C:\Users\LabVIEW-user\AppData\Local\Temp\GmailAttachmentSender.vi Last modified on 5/20/2016 at 6:16 PM Printed on 5/20/2016 at 6:16 PM

GmailAttachmentSender.vi



Outgoing Mail Server (SMTP)	Sender's Email Address (eg: LabVIEW@	gmail.com) Sender's Name
GMAIL Account User ID	Recipient's Email Address	Recipient's Name
GMAIL Account Password	2nd Recipient's Email Address 2	
	Subject	
Port	Body	
÷		
error in (no error)		
source		
error out	Attachment 1	
status code	8	
✓ d0		
source	Attachment 2	
	8	

Page 2

GmailAttachmentSender.vi C:\Users\LabVIEW-user\AppData\Local\Temp\GmailAttachmentSender.vi Last modified on 5/20/2016 at 6:16 PM Printed on 5/20/2016 at 6:16 PM





Appendix 43 Header Gen (SubVI) version 2 - run logger attempt to add

comments.vi



Header Gen (SubVI) version 2 - run logger attempt to add comments.vi C:\Users\LabVIEW-user\AppData\Local\Temp\Header Gen (SubVI) version 2 - run logger attempt to add comments.vi Last modified on 5/20/2016 at 6:17 PM

Printed on 5/20/2016 at 6:19 PM

Header Gen (SubVI) version 2 - run logger attempt to add comments.vi



Appendix 44Periodic Fast Sweeps Temp Dependent v2b SubVI load with

callers Default.vi



Periodic Fast Sweeps Temp Dependent v2b SubVI load with callers Default.vi C:\Users\LabVIEW-user\AppData\Local\Temp\Periodic Fast Sweeps Temp Dependent v2b SubVI load with callers Default.vi Last modified on 5/20/2016 at 6:19 PM Printed on 5/20/2016 at 6:20 PM

Periodic Fast Sweeps Temp Dependent v2b SubVI load with callers Default.vi

Number of Big Steps * (Sample Clock Rate /10) = Total # of Ste	ps for Output	
Max Output Voltage * 2 / Total # of Steps = Step Height in V		
Sample Clock Rate (in Hz)	Current Temp	Next Temp to take a measurement
Start Voltage Number of Big steps Resistor Value		
Sample #		
153		
Layer #		
No Magnetic Field Incremental degrees in K to take IV sweep (default: 5K)		
Operator		
Kiar		
Comments		



Periodic Fast Sweeps Temp Dependent v2b SubVI load with callers Default.vi C:\Users\LabVIEW-user\AppData\Local\Temp\Periodic Fast Sweeps Temp Dependent v2b SubVI load with callers Default.vi Last modified on 5/20/2016 at 6:19 PM Printed on 5/20/2016 at 6:20 PM





Time Delay

Inserts a time delay into the calling VI. -----

This Express VI is configured as follows:

Delay Time: 20 s

Appendix 45 Periodic Sweeps Temp Dependent v4c - Neg to Pos - Num runs

calculated.vi



Periodic Sweeps Temp Dependent v4c - Neg to Pos - Num runs calculated.vi C:\Users\LabVIEW-user\AppData\Local\Temp\Periodic Sweeps Temp Dependent v4c - Neg to Pos -Num runs calculated.vi Last modified on 5/20/2016 at 6:21 PM Printed on 5/20/2016 at 6:23 PM

Periodic Sweeps Temp Dependent v4c - Neg to Pos - Num runs calculated.vi

6				
		Current Temp	Next Temp to take a me	asurement
Sample #				
153				
Layer #				
004				
Incremental degrees in K to take IV sweep	(default: 25K)			
7 10				
Operator Kiar				
Community .				
Comments				
Γ				
				00000
				┝━┥│
		Connection (Connection)	9	



Periodic Sweeps Temp Dependent v4c - Neg to Pos - Num runs calculated.vi C:\Users\LabVIEW-user\AppData\Local\Temp\Periodic Sweeps Temp Dependent v4c - Neg to Pos -Num runs calculated.vi Last modified on 5/20/2016 at 6:21 PM Printed on 5/20/2016 at 6:23 PM





Time Delay

Time Delay Inserts a time delay into the calling VI.

This Express VI is configured as follows:

Delay Time: 20 s

Appendix 46 RunCalculatorINI.vi

Page 1

RUN NUM

RunCalculatorINI.vi C:\Users\LabVIEW-user\AppData\Local\Temp\RunCalculatorINI.vi Last modified on 5/20/2016 at 6:24 PM Printed on 5/20/2016 at 6:24 PM

RunCalculatorINI.vi





Appendix 47 RunInfoLogger.vi

Page 1 RUN INFO LOGGER

RunInfoLogger.vi C:\Users\LabVIEW-user\AppData\Local\Temp\RunInfoLogger.vi Last modified on 5/20/2016 at 6:24 PM Printed on 5/20/2016 at 6:25 PM

RunInfoLogger.vi

Run Info	RUN INFO LOGGER
Run Info	

C:\Users\LabVIEW-user\Documents\My Programs\Delta Measure Testing\Test Measurements\RunInfo_Log.txt



Appendix 48 SubVI for R v T run v3 - Case structure removed.vi



Sub VI for R v T run v3 - Case structure removed.vi







Page 3	
	- 3



Convert from Dynamic Data

Converts the dynamic data type to numeric, Boolean, waveform, and array data types for use with other VIs and functions.





Appendix 49 Sweep Up and Down v5e - Neg to Pos - MF selection and

CryoState added.vi



Sweep Up and Down v5e - Neg to Pos - MF selection and CryoState added..vi C:\Users\LabVIEW-user\AppData\Local\Temp\Sweep Up and Down v5e - Neg to Pos - MF selection and CryoState added..vi Last modified on 5/20/2016 at 6:27 PM Printed on 5/20/2016 at 6:28 PM

Sweep Up and Down v5e - Neg to Pos - MF selection and CryoState added..vi

Magnetic Field —	
Sample #	
Layer # 🚽 🗖	
Cryo State 🖵	
Operator	
Comments	

Calculates the current from the total number of steps.



Sweep Up and Down v5e - Neg to Pos - MF selection and CryoState added..vi C:\Users\LabVIEW-user\AppData\Local\Temp\Sweep Up and Down v5e - Neg to Pos - MF selection and CryoState added..vi Last modified on 5/20/2016 at 6:27 PM Printed on 5/20/2016 at 6:28 PM





Sweep Up and Down v5e - Neg to Pos - MF selection and CryoState added..vi C:\Users\LabVIEW-user\AppData\Local\Temp\Sweep Up and Down v5e - Neg to Pos - MF selection and CryoState added..vi Last modified on 5/20/2016 at 6:27 PM Printed on 5/20/2016 at 6:28 PM





Build XY Graph Build XY Graph

formats the data displayed on an X-Y Graph.

Page 4

Sweep Up and Down v5e - Neg to Pos - MF selection and CryoState added..vi C:\Users\LabVIEW-user\AppData\Local\Temp\Sweep Up and Down v5e - Neg to Pos - MF selection and CryoState added..vi Last modified on 5/20/2016 at 6:27 PM Printed on 5/20/2016 at 6:28 PM



Appendix 50 TextMessageGeneral.vi



TextMessageGeneral.vi C:\Users\LabVIEW-user\AppData\Local\Temp\TextMessageGeneral.vi Last modified on 5/20/2016 at 6:28 PM Printed on 5/20/2016 at 6:29 PM

TextMessageGeneral.vi





Appendix 51 Voltage reading with DC current always on v4 Compressor

Automatically on and off.vi



Voltage reading with DC current always on v4 Compressor Automatically on and off.vi C:\Users\LabVIEW-user\AppData\Local\Temp\Voltage reading with DC current always on v4 Compressor Automatically on and off.vi Last modified on 5/20/2016 at 6:29 PM Printed on 5/20/2016 at 6:30 PM

Voltage reading with DC current always on v4 Compressor Automatically on and off.vi



Page 2

Voltage reading with DC current always on v4 Compressor Automatically on and off.vi C:\Users\LabVIEW-user\AppData\Local\Temp\Voltage reading with DC current always on v4 Compressor Automatically on and off.vi Last modified on 5/20/2016 at 6:29 PM Printed on 5/20/2016 at 6:30 PM





Voltage reading with DC current always on v4 Compressor Automatically on and off.vi C:\Users\LabVIEW-user\AppData\Local\Temp\Voltage reading with DC current always on v4 Compressor Automatically on and off.vi Last modified on 5/20/2016 at 6:29 PM Printed on 5/20/2016 at 6:30 PM





Build XY Graph Build XY Graph formats the data displayed on an X-Y Graph.

Page 4

Voltage reading with DC current always on v4 Compressor Automatically on and off.vi C:\Users\LabVIEW-user\AppData\Local\Temp\Voltage reading with DC current always on v4 Compressor Automatically on and off.vi Last modified on 5/20/2016 at 6:29 PM Printed on 5/20/2016 at 6:30 PM





Voltage reading with DC current always on v4 Compressor Automatically on and off.vi C:\Users\LabVIEW-user\AppData\Local\Temp\Voltage reading with DC current always on v4 Compressor Automatically on and off.vi Last modified on 5/20/2016 at 6:29 PM Printed on 5/20/2016 at 6:30 PM



Time Delay Time Delay

Inserts a time delay into the calling VI.

This Express VI is configured as follows:

Delay Time: 5 s

VITA

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PUBLICATIONS

Larkins, G., Vlasov, Y. and Holland, K. (2015) *Evidence of Superconductivity in Doped Graphite and Graphene*. Superconductor Science and Technology, Volume 29, Number 1.