RESISTIVITY CHANGES IN

CARBON-IMPLANTED TEFLON

A Senior Honors Thesis

by

MATTHEW R. JACKSON

Submitted to the Office of Honors Programs
& Academic Scholarships
Texas A&M University
In partial fulfillment of the requirements of the

UNIVERSITY UNDERGRADUATE
RESEARCH FELLOW

April 2004

Major: Nuclear Engineering

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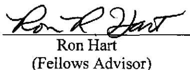

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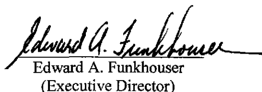

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ABSTRACT

Resistivity Changes in Carbon-implanted Teflon (April 2004)

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Department of Nuclear Engineering

The change in resistance of Teflon due to the implantation of carbon atoms was measured. The procedure involved implanting carbon at energies of 40 kV, 50 kV, and 140 kV using beam currents ranging from 0.5 μA to 3 μA for time intervals ranging from 30 minutes to over an hour. Silver paste was used to attach leads to the implanted Teflon samples, and a Fluke multimeter utilized to measure the voltage across a 10 MΩ resistor placed in series with the Teflon-implanted resistor. Significant deviations from Teflon's native resistance were observed and exhibited exponential decreases in resistance with increasing voltage.

ACKNOWLEDGMENTS

I want to thank Dr. Hart for his direction and encouragement during the past year. I would like to thank Don Algama, Avery Bingham, Francois Clement, and Trey Johansson for their help during experiment preparation. I want to extend my sincerest thanks to Lucas Phinney for his patience and willingness to help me operate the accelerator during my first two experiments. Finally, I would like to thank the Honors Department for the opportunity to participate in the Fellows program and for the fiscal support provided by the College of Engineering's Undergraduate Summer Research Grant.

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CHAPTER I

INTRODUCTION

Motivation

Dr. Ron R. Hart of Texas A&M's Nuclear Engineering Department is currently investigating the direct conversion of fission fragments into electrical energy. It is estimated that this process (discussed later in detail) will produce 4MV of electrical voltage. In order to test the direct conversion process, it is necessary to use a high energy accelerator, in this case, TAMU's Cyclotron.

The Cyclotron only provides a current of 100 nA, thus to produce 4MV, a resistance on the order of 10^{13} Ω is required to stabilize the voltage. The purpose of this study was to determine the feasibility of creating such a high resistance resistor through implanting a conductive material into an insulator; namely implanting conductive carbon atoms into Teflon.

Current Progress in Direct-conversion Research

The direct conversion of fission fragments to energy (DCFFE) is not a new idea – the concept was originally proposed by E.P. Wigner in 1944. In 1957 G. Safonov performed the first theoretical study. The early experiments yielded poor results, and largely due to the technical limitations of the period, most DCFFE research was ended by the late 1960s.[1]

In recent years there has been a renewed interest in this subject. Today, most DCFFE research is supported through the Department of Energy Nuclear Energy

Research Initiative's Direct Energy Conversion Project[2]. Preliminary analysis suggested that the use of a fission fragment magnetic collimator reactor (FFMCR) could offer promising results[3].

Dr. Hart is currently conducting research in the area of out-of-core direct fission fragment energy conversion utilizing a magnetic collimator. Unlike traditional direct energy conversion (DEC) systems that reside in the reactor core region, the out-of core approach involves four main components:

- Nuclear reactor core consisting of fuel elements with ultra-thin fuel layers;
- Central solenoid (CS);
- Conical magnetic collimators (CMC);
- Multi-stage direct energy collectors.

One advantage of this design is that the electromagnetic equipment is located outside the strong radiation environment of the core. Figure 1 (Following page) shows the principal components of the proposed FFMCR system. The core design is similar to the one proposed by Chapline for a fission fragment rocket[4]. The basic power source is the kinetic energy of the fission fragments that escape from an extremely thin fuel layer. The core is designed in such a way as to allow the fission fragments to be magnetically guided to out-of-core collectors. The collector decelerates the FFs, producing a large positive voltage. As stated earlier, the voltage produced by this process is expected to be on the order of 4 MV.

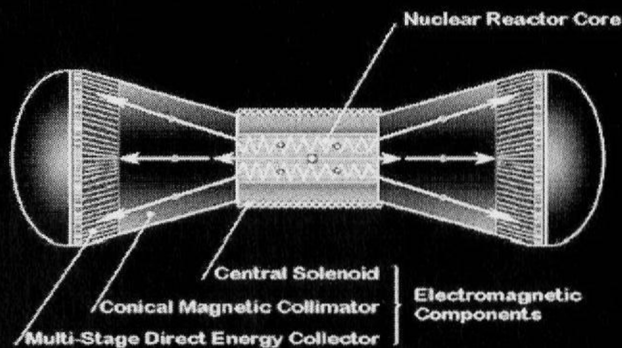


Figure 1: Principal Component Arrangement of the FFMCR System

History of Ion Implantation

Since it is proposed to use ion implantation into an insulator to assist current research in direct conversion of fission fragments to electricity, it is helpful to summarize the milestones achieved in ion implantation science. Ion implantation is a broad topic with innumerable applications. The first recorded attempt to artificially implant ions in a target was accomplished by Rutherford in 1906 when he bombarded an aluminum plate with alpha particles[5]. However the pioneer of this process was not concerned with the structural change in the target, but rather with the backscattering of alpha particles from the plate. The first reports of ion bombardment in semiconductors occurred in 1948, but until 1954 no operative device was manufactured. In 1954 Shockley patented the use of ion beams for producing the buried base layer in a bipolar transistor. Hence, ion implantation was introduced as an alternative to diffusion in forming junctions in the transistor. The critics scoffed at Shockley's results, dubbing ion implantation "a sophisticated, expensive yet brutal method for simply doping delicate

semiconductors". Despite these criticisms, a large number of laboratories began researching ion implantation in the early sixties.

Many of the accelerators and separators used in nuclear research could not keep up with the increasing energy trends, and new uses had to be found for them. Thus the equipment needed for the research of ion implantation already existed and served as valuable tools until better implanters were designed to fit the needs of researchers. The problems with these accelerators were their high energy and low current. Thus they were poorly suited for ion implantation of semiconductors and led researchers to develop their own implanters. The major breakthrough in the industrial use of ion implanters to dope silicon occurred in 1966, when Hughes Research Laboratory developed the "Ultra High Vacuum Implanter", and within two years later the same laboratory produced implanters approaching industrial class. In 1973 the first true industrial implanters were manufactured. As recently as 2003, ion implantation was used to modify the resistance of diamond by four orders of magnitude.[6]

CHAPTER II

EXPERIMENTAL

Overview

C^+ atoms were implanted into a circular Teflon target utilizing ionized CO_2 gas in a 150 kV accelerator – mass spectrum analysis was performed to determine the magnetic field value corresponding to a carbon beam. The target was housed within a Faraday cup within the high-vacuum target chamber. The change in resistance of the implanted Teflon target was measured using a Fluke multimeter and a 10 kV power supply.

Accelerator System

The accelerator consists of eight primary components: the ion source, the linear acceleration column, the glass cross, a mass separation magnet, knife edge collimators, an ion pump, a beam profiler, and the target chamber [Figure 2].

Gas enters the accelerator and becomes ionized by the tungsten-plasma filament [L]. The ionized atoms are then accelerated through a linear acceleration column under high vacuum to energies up to 140 kV [K]. Next, the ions pass through a glass cross that contains deflection plates, a shutter, and a 1" diameter aperture designed to prevent electron back streaming into the source [J]. A diffusion pump is attached to the bottom of the glass cross, and maintains a vacuum operating at approximately 10^{-6} torr. After the ions pass through the glass cross they enter a disk magnet with a 20 degree bend [I].

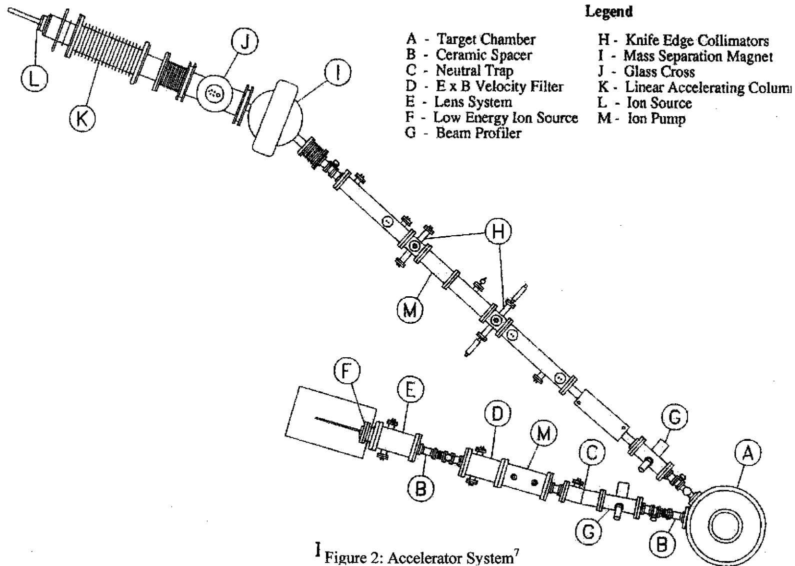


Figure 2: Accelerator System[7]

After the ions pass through the magnet, they pass through a series of knife edge collimators [H] that can be adjusted to improve beam uniformity. Finally, the ions pass through a beam profiler [G] that outputs the shape of the beam on an oscilloscope and then through a final collimator into the target chamber.

Target Chamber

The target chamber vacuum is maintained by both a cryopump and a diffusion pump. The Faraday cup within the target chamber rests on a goniometer, a device that controls the precise translation and rotation of the target. The target used for the experiment was a 1.5 inch diameter Teflon target (thickness $\approx$ 0.25 inch). The target was placed inside a Faraday cup [Figure 3] and an electron filament attached to the surface of the cup. The electron filament was constructed from a Sylvania 7880 automotive lamp. The glass bulb was first cut to expose the tungsten filament. Teflon coated wires were then soldered to the bulb and the bulb attached to feed-through connections inside the target chamber.

Mass Spectrum

The first step was to experimentally determine the magnetic field strength corresponding to a carbon beam. To accomplish this, the accelerator was operated at a voltage of 140 kV, the magnetic field strength was gradually increased and each time a spike in the current was encountered, the corresponding magnetic field was recorded and the ion properly identified [Figure 4].

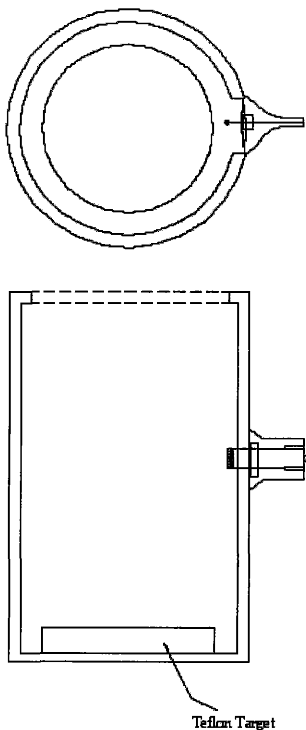


Figure 3: Target/Filament Assembly

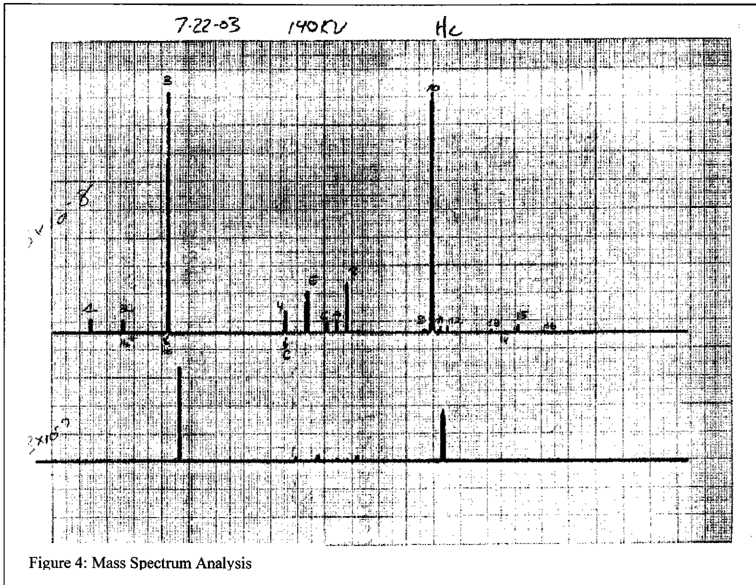


Figure 4: Mass Spectrum Analysis

A magnetic field of 0.2551 T was determined to produce carbon at 140 kV. This value was modified for other beam energies according to equation 1. Equation 1 is derived from equating the centripetal force with the force of magnetic field acting on a charged particle.

$$\frac{B_{140kV}}{B_{new}} = \sqrt{\frac{140}{E_{new}}} \qquad (1)$$

Electron Filament

Implanting ions into an insulating target eventually leads to buildup of large voltages on the target surface, eventually repelling any ions incident on its surface and preventing further implantation into the target. To avoid this, the electron filament in figure 3 was constructed. However, it was also necessary to obtain an estimate of the electron flux from the filament to determine the filament voltage at which sufficient electrons are present to cancel out any excess positive charge. To accomplish this, the circuit pictured in figure 5 was constructed. A negative bias of 100 V was applied to the filament to ensure all electrons were attracted to the surface of the faraday cup. The ammeter then measured the current to ground. Voltages were adjusted from zero to seven volts and the corresponding current recorded. The results of this experiment are plotted in figure 6. It was determined that operating the filament at 6.5 volts would provide an electron flux sufficient to prevent charge buildup.

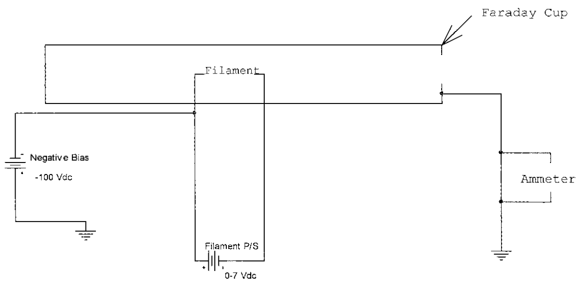


Figure 5: Filament Test Circuit

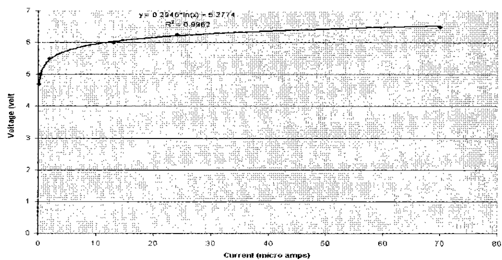


Figure 6: Filament Voltage vs Emission Current

Target Implantation

To manufacture the Teflon resistor, a 0.75 inch-long implant was made. For each experiment an 1/8" collimator aperture was utilized. The goniometer was used to translate the target over the requisite 0.75 inch length.

Target Pre- and Post-Implantation Treatment

Prior to implantation the target's surface was cut using a lathe. Next it was cleaned with acetone to remove any contaminates. Finally, the target was cleaned with methanol to remove any residue left by acetone. The target was placed inside the target holder while wearing latex gloves and then immediately placed inside the target. Pump down was then initiated. Following implantation the target was carefully removed from the target holder, gloves again being used to reduce target contamination. Silver paste was utilized to attach two Teflon-coated wires to the edge of each implant [Figure 7] and the paste was allowed to cure over a 24 hour period.

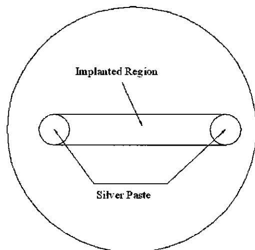


Figure 7: Post-Implant Target Preparation

Resistivity Measurement

The carbon-implanted Teflon resistor was placed in series with a 10 MΩ resistor. A Fluke multimeter with sensitivity of 0.01 mV was placed in parallel with the 10 MΩ resistor and the voltage across the 10 MΩ resistor measured [Figure 8]. The power source utilized was a 10 kV, NIST compliant, DC power supply.

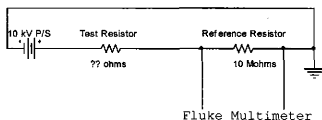


Figure 8: Resistor Test Circuit

CHAPTER III

RESULTS AND DISCUSSION

Beam Current Stability

The beam current exhibited stability issues when operating at low energies (40-50 kV). Consequently, two choices were available; first, to implant the carbon in one continuous layer, while allowing the beam current to decrease with time or two, to implant the carbon for a short interval, stop the translation of the target, readjust the beam to restore the original current. The problem with the first option is that the density of implanted carbon is lower (by a factor of 2) at the end of the implanted length than at the beginning. On the other hand, if option 2 is chosen, a discontinuity in the implanted region will occur due to the necessity of stopping the beam. In experiments I and III option 1 was chosen, while option 2 was used during experiment II.

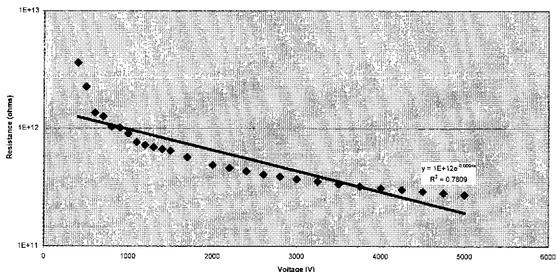
Experiments I-II

Experiments I-III shared approximately the same operating conditions. In each case the vacuum in the target chamber was in the 10^{-7}-10^{-8} torr range, the vacuum in the beam line was on the order of 10^{-8} torr, and the vacuum pressure at the glass cross was on the order of 10^{-6} torr.

Experiment I was conducted in October 2003. Carbon atoms were implanted with a beam current ranging from 0.9 μA at the beginning to 0.4 μA at the end at an energy of 50 kV over the 0.75 inch length during a 30 minute interval, with the beam remaining stationary on each end for 5 minutes to ensure a dense layer of carbon to

adhere to the leads. The implant was visually identifiable by a yellowish discolored line at the center of the target. Silver paste was then applied to the target and the resistance measured. The results are shown in figure 9. The resistance decreased a full order of magnitude over the 0-10 kV region. After 5 kV the voltage began to fluctuate rapidly, indicating that the current was shorting across the resistor surface. It was reasoned that this was caused by surface contamination. Irregularities from the general exponential trend at energies below 1 kV can be attributed to the sensitivity limits of the Fluke multimeter.

Figure 9: Experiment I Resistance Plot



During experiment II carbon atoms were implanted into Teflon at 40 kV over a 45 minute time interval, with the beam concentrated at the end points for 10 minutes/point. The beam current for this implant was maintained at 0.5 μA, stopping the implant after 20 minutes to re-calibrate the beam. The target was then removed, silver

paste applied, and the resistance measured. No resistance was measurable. It was reasoned that this could be due to one of two causes: first, no resistance was measured because there were contaminates on the target surface, and that the resistance change measured in experiment I was due to surface contamination, or secondly, that no resistance was measured due to a discontinuity in the implant – preventing conduction across the carbon implanted region.

Experiment III

Since experiment III was to be the final test, great care was taken to maximize the chance of success. The depth ions are implanted in a material increases as the energy of the incident beam is increased. Around this average depth a Gaussian distribution emerges. This tends to be broadened slightly through sputtering, or the removal of surface ions through their impact with the incident ions. This results in several monolayers of erosion in the surface thickness – this erosion was visually observable when examining the implanted target of experiment III.

Resistance is a function of the length, width, and depth of the implant. During the final experiment it was resolved to increase the depth of the implant. It was considered that if carbon were implanted over the same region at two different energies, overlapping Gaussian curves would be produced, leading to an increase in the depth-thickness of the implant. Figures 10 and 11 illustrate the variation in depth-thickness expected to be achieved by implanting at multiple energies. The average range and standard deviation for each energy was obtained using TRIM[8] tables for carbon implanted into Teflon. A MATLAB code was developed that inputted the respective

ranges and standard deviations for the energies, and calculated the Gaussian carbon distributions over a fixed spatial region for the length of time the beam was expected to remain over a target region. The sputtering yield was estimated to be 2 Teflon atoms per incident C atom, and it was assumed that the beam was perfectly uniform. A comparison of the plots shows that the implantation thickness is increased by more than a factor of two by using dual implantation energies versus a single implantation at 50 kV.

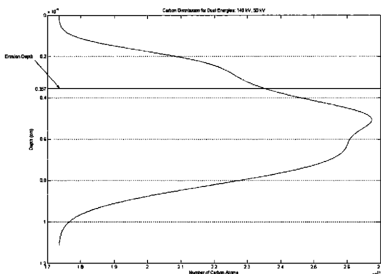


Figure 10: Carbon Distribution vs Depth for 50 kV/140 kV Dual Implantation

Based on the simulation results above, it was determined that using dual energies would maximize the concentration of carbon in the implanted area. Consequently carbon was implanted over 110 minute intervals each at energies of 50 kV and 140 kV. For the 50 kV implant, the current varied from 1.1 μA initially to a final current of 0.8 μA.

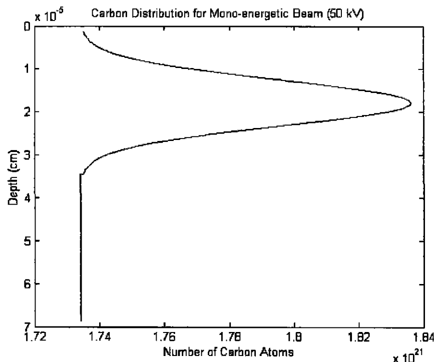
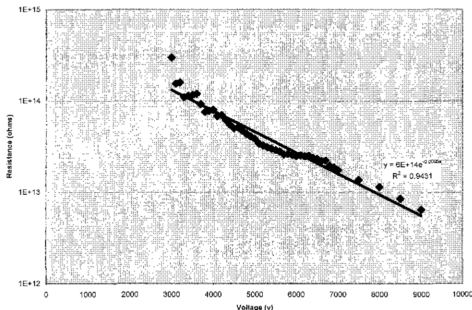


Figure 11: Carbon Distribution vs Depth for 50 kV Implantation

During the 140 kV implant 3 μA of beam current was obtained, and finished with a 2.9 μA current. The beam remained stationary over each endpoint for 15 minutes at an energy of 50 kV to ensure ample carbon density for silver contacts. A visual examination of the target differentiated it from the target in experiment I. A yellowish-ring existed around the implanted region and the region in the center was white, apparently caused by surface erosion. The significant surface erosion was predicted by computer simulation, just as the surface erosion was accurately predicted by the simulation to be negligible in the case of the 50 kV implant. This seems to indicate that the surface discoloration of experiment I may have been due to radiation damage rather than the presence of carbon.

The target was removed and silver paste used to attach leads to the surface, and the resistance measured. Figure 12 shows the trend observed. The spikes in the resistance are most likely due to sensitivity issues with the Fluke multimeter. At the range of 3-4 kV the voltage read between 0.01 and 0.02, testing the limits of the meter's sensitivity. As in experiment I, an exponential decrease in the resistance was observed, with a minimum resistance on the order of 10^{13} Ω observed. After 9500 volts the current

Figure 12: Experiment III Resistance Curve



again seemed to short across the Teflon resistor, indicating that perhaps some contamination existed on the Teflon's surface. To verify that this was not solely the product of surface contamination, silver paste was placed at a distance of approximately 0.5 inch from the implanted Teflon, and the resistance measured. No resistance was recorded until 9000 V, at which point the voltage read by the multimeter oscillated so rapidly as to make it impossible to record a value with any degree of confidence. This

seems to indicate that this change was induced by implantation and secondly that the exponential decrease in the first experiment was not entirely due to surface contamination, and that perhaps the surface contamination only caused a linear decrease from the resistances produced in the third experiment.

Additionally, this exponential decrease seems to be supported by the recorded activation energies. Theory would predict that the activation energy for the lower density implant be higher than that of a denser implant, as the lower density would tend to be more resistant to electrical flow. The significant difference in the resistance appears then to be the result of differences in initial resistances, the difference in which could be attributed to the presence of surface contamination on the target of experiment I.

CHAPTER IV

CONCLUSIONS AND FUTURE WORK

Conclusions

It appears that implanting carbon into Teflon does produce significant changes in resistance. It appears that the resistance does decrease exponentially. The results of experiment I also show that the resistance is extremely sensitive to even small amounts of surface contamination – to such an extent that might warrant more extraordinary means of cleaning the target than acetone and methanol.

Initial results still leave the source of the resistance change in doubt. It is unclear whether the change is due to the addition of conductive carbon, or merely the result of extensive radiation damage. Since Teflon is C_2F_4 there is a chance that the radiation damage causes some of the carbon atoms to align with one another, producing the resistance change observed in experiments I and III.

Finally, it was shown that the MATLAB code used to simulate the implantation profile produced reasonably accurate results, at least from visible inspections of the respective targets.

Future Work

The results of this research have opened several possible avenues for further study. First, by implanting a non-conductive ion, such as neon, one could determine whether the change in resistance was due to radiation damage or to the addition or carbon. Second, one might change the target material from Teflon to a ceramic material

and establish whether carbon implanted into a ceramic produces a similar V-R curve. Finally, one could try annealing the target to find out if this changes the behavior of the carbon-implanted Teflon.

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APPENDIX I

MATLAB IONRANGE SOURCE CODE

```
%NOTE: rp1 must be lower energy of 2 implantations, entered in angstroms
function ionrange(rp1,rp2,s1,s2,t1,t2,l,d,C)
%rp1,rp2, median range of respective implantations (from srim,in angstrom)
%s1,s2 standard deviation of respective implantations (from srim, in angstrom)
%t1,t2 time over which sample is implanted
%l=length of sample (cm)
%d=collimater aperature diameter (in)
%C=beam current, amps
%mm=molar mass g/mol
%Na=avagadro's number atoms/mol
%rho=density of target, g/cc
mm=100;
Na=6.022e+23;
rho=2.2;
rp1=rp1*1e-8; %changing units of rp1 to cm
rp2=rp2*1e-8; %changing units of rp2 to cm
s1=s1*1e-8; %changing units of s1 to cm
s2=s2*1e-8; %changing units of s2 to cm
dt=0.1; %setting a static time step of 0.1 sec
d=2.54*d; %changing units to cm
tirr1=d/l*t1; %irradiation time over differetial area in sec
tirr2=d/l*t2; %irradiation time over differetial area in sec
deltax=(rp2+3*s2+3.5702e-005)/1000; %setting dx such that the entire range will be
%calculated within a thousand steps
flux=C/(pi*d^2/4)/(1.6022e-19); %beam flux in n/(cm^2-sec)

y1 = zeros(1,1001); %pure carbon distribution for first implantation
y2 = zeros(1,1001); %pure carbon distribution for second implantation
y = zeros(1,1001); %total pure carbon distribution for two implantations


tsteps1=tirr1/dt; %number of time steps for first implantation
tsteps2=tirr2/dt; %number of time steps for second implantation
dxtot=(tirr1+tirr2)*2*flux/(1/mm*Na*rho);
i=0;
for t=0:dt:tsteps1
i=i+1;

if (i==1)
   dx=0;
```

```
else
   dx=dx+2*flux*dt/(1/mm*Na*rho);  %assuming 2 atoms removed per ion in
end

j=0;

if (i==1)
   for x=0:deltax:rp2+3*s2+dxtot;
   j=j+1;
   y1(1,j)=3/7*C*dt*exp(-(x-(rp1+dx))^2/(2*s1^2))/(1.6022e-19);  %pure carbon
%concentration neglecting carbon present in PTFE
   end
else
   j=0;

   for x=0:deltax:rp2+3*s2+dxtot;
   j=j+1;
   y1(1,j)=3/7*C*dt*exp(-(x-(rp1+dx))^2/(2*s1^2))/(1.6022e-19)+y1(1,j);  %pure
%carbon concentration neglecting carbon present in PTFE

   end
end
end
i=0;

for t=0:dt:tsteps2
i=i+1;

dx=dx+2*flux*dt/(1/mm*Na*rho); %assuming 2 atoms removed per ion in

j=0;

if (i==1)
   for x=0:deltax:rp2+3*s2+dxtot;
   j=j+1;
   y2(1,j)=3/7*C*dt*exp(-(x-(rp2+dx))^2/(2*s2^2))/(1.6022e-19);  %pure carbon
%concentration neglecting carbon present in PTFE
end
else
   for x=0:deltax:rp2+3*s2+dxtot;
   j=j+1;
   y2(1,j)=3/7*C*dt*exp(-(x-(rp2+dx))^2/(2*s2^2))/(1.6022e-19)+y2(1,j); %pure carbon
% concentration neglecting carbon present in PTFE

   end
```

```
end
end
imax=i;
x(1)=0;
for i=1:1000
   xcoor(i+1)=deltax*i;
end

dx

for j=1:1001
y(1,j)=((y1(1,j)+y2(1,j))+.24/100*1.2*6.022e+23*((3*s2+rp2)*pi*d^2/4))/((3*s2+rp2)*
pi*d^2/4);
end
i=1;

while (xcoor(1,i)<dx)
   i=i+1;
   g=i;
end
i
if (i==0)
fprintf('Warning, solution did not converge')
i=1;
end
xval=zeros(1,1001-(i-1));
yval=zeros(1,1001-(i-1));
for j=1:1001-(i-1)
yval(1,j)=y(1,i-1+j);
xval(1,j)=xcoor(1,i-1+j);
end

plot (yval(1,:),xval(1,:));
```

VITA

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