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Performance of Nanomaterials and Actively Running Nanocircuits During Heavy Ion Irradiation

H.C. Shaw[1], D. Liu[1], B.W. Jacobs[2], V.M. Ayres[2], R.M. Ronningen[3], A.F. Zeller[3], M.A. Crimp[2], J. Halpern[4], M-Q He[4], G.L. Harris[4], and M.P. Petkov[5]

[2]NASA Goddard Space Flight Center, Code 562, Greenbelt, MD 20771

[1]College of Engineering, Michigan State University, East Lansing, 48824

[3]NSCL Laboratory, Michigan State University, East Lansing, MI 48824

[4]Materials Research Center of Excellence, Howard University, Washington DC, 20059

Abstract

The development of nanocircuits for space applications is an emerging technology area for NASA. We will preset our recent research results on the performance of both nanomaterials and actively running nanocircuits during heavy ion irradiation. The radiation experiments were performed at the National Superconducting Cyclotron Laboratory at Michigan State University whose available primary beams and beam energies well match the energy spectra of abundant charged particles in galactic cosmic rays. Primary beams of Krypton having mass numbers of 78 and 86 respectively were used in two sets of experiments, representative of the high-Z heavy ions encountered in a space radiation environment.

Introduction

Energetic heavy ions having many-tens to hundreds of MeV per nucleon are a serious source of non-recoverable electronic upsets for satellites. They are highly penetrating and capable of generating penetrating secondary ions in all but the most massive radiation shielding. As the increased use of miniaturized equipment corresponds to a decreased availability to carry any shielding, radiation damage by heavy ions will become an increasing challenge.

The development of nanocircuits for space applications is an emerging technology area for NASA. We will present our recent research results on the performance of both nanomaterials and actively running nanocircuits during heavy ion irradiation. The radiation experiments were performed at the National Superconducting Cyclotron Laboratory at Michigan State University. The National Superconducting Cyclotron Laboratory is particularly suited for such studies because its available beam energies well match the energy spectra of abundant charged particles in galactic cosmic rays. Primary beams of Krypton having mass numbers of 78 and 86 respectively were used in two sets of experiments, representative of the high-Z heavy ions encountered in a space radiation environment.

In one set of experiments gallium nitride (GaN) nanowires and nanocircuits were irradiated using a primary beam of Krypton having mass number 78 (^{78}Kr) at 140.32 MeV per nucleon (MeV/u), delivered by the coupled cyclotron facility at the National Superconducting Cyclotron Laboratory (NSCL) The experiments were performed in the NSCL S1 Vault Single Event Excitation Test Facility (SEETF) Vault end station, which is specially equipped with electronic and beam operations connections to a remote control room[1]. This enabled the first real-time characterization of the electronic performance of a GaN-based field effect transistor (FET) during ^{78}Kr irradiation. Irradiations of GaN nanowires were also carried out separately under the same beam conditions.

In another set of experiments, carbon nanomaterials including single and multi wall carbon nanotubes and electrospun carbon nanofibers, with well-graphitized vapor grown carbon fibers as controls, were irradiated using a primary beam of Krypton having mass number 86 (^{86}Kr) at 142A MeV. All of these nanomaterials are hollow care graphitic structures with varying wall structures.

Experimental Results

(1) Summary of Real -time Electronic Performance of Irradiated GaN Nanocircuit

We will summarize real-time GaN nanocircuit performance during irradiation previously reported in References (**Error! Bookmark not defined.**,2) to show the potential that nanowire based devices may have in radiation environments. A GaN nanowire FET design using an n-type semiconducting channel[3] was used in the radiation experiments (Figure 1). Nanotube/nanowire FET designs achieve transistor functionality via unconventional Schottky barrier modulation at the contacts rather than by standard channel modulation[4,5]. The nanowire was connected to contacts pads using electron beam lithography and wire-bonded to a dual-in-line package.. The I-V characteristics of the FET were taken before, during and after irradiation with different gate-source voltages (V_{GS}) and swept source-drain voltages (V_{SD}), using a Keithley 487 Picoammeter/Voltage Source and a Hewlett Packard 6633A System DC Power Supply. Measured pre radiation I-V characteristics at 0, 3, 6, and 9 Volts V_{GS} demonstrated that the nanowire was n-type and its conduction could be altered by varying V_{GS}.

Real time electronic measurements during ^{78}Kr heavy ion irradiation were implemented in LabVIEW[6] using a remote-connection computer during the experiments. All measurements were made in air at room temperature. Irradiation was initiated at a low level intensity of 10^2 particles per second (pps), and subsequently increased to 10^6 pps with intermediate intensities of 10^4 pps and 10^5 pps. The gate voltage was also increased through 0, 3, 6, and 9 Volts. Each combination of conditions was maintained for 600 seconds, therefore the beam fluence per run ranged from 3×10^4 to 3×10^7 particles/cm^2. The selected radiation levels corresponded to ones that have caused electronic upsets in conventional circuits during ^{78}Kr irradiation[7] and were the highest available at the SEETF facility.

The real time I-V characteristics of the active GaN nanowire FET during ^{78}Kr irradiation are shown in Figure 2(d). The final active nanocircuit conditions were 9 Volts V_{GS}, 0-22 Volts V_{SD}, under 5×10^5 particles/sec-cm^2. The results indicated normal real-time electronic function. At the conclusion of the radiation experiments, after exposure to a cumulative fluence of 3.30×10^7

particles/ cm^2, the post-radiation electronic performance of the GaN FET was again measured, first at 9 Volts V_{GS} followed by 12 Volts V_{GS}. Normal electronic function was observed under these post-irradiation high bias conditions.

(2) Pre- and Post Irradiation Characterization of GaN Nanowires

The stable performance of the GaN FET nanocircuit is determined by both the nanowire properties and the nanocircuit architecture. The GaN nanowires were grown in a direct reaction of metal gallium vapor with flowing ammonia at 850-900°C without a catalyst, resulting in diameters of ~50-100 nm[8,9]. They displayed a new two-phase coaxial crystalline homostructure with an inner wurtzite phase crystal structure and a coaxial outer zinc-blende phase crystal structure, reported in Reference (10)..The inner wurtzite phase diameter was typically 30 to 40 nm and the outer zinc-blende layer was 20 to 60 nm, with an overall nanowire diameter of 50 to 100 nm. High resolution transmission electron microscopy (JEOL FS2200) with nanodiffraction, selected area electron diffraction, energy dispersive spectroscopy, electron energy loss spectroscopy, and fast Fourier transforms, of over thirty GaN nanowires grown under the above conditions was used to identify the two crystalline phases.

HRTEM images of the GaN nanowires are shown in Figure 3(a-b). Most GaN nanowires displayed the smooth and highly crystalline inner core and outer layer, with a sharp ~1-3 atomic layer interface, shown in Figure 3(a). However GaN nanowires with irregular outer layers were sometimes observed, as shown in Figure 3(b).

We summarize post-irradiation results previously reported in Reference ([11]). In the nanomaterials experiments, a volume of material was prepared and weighed using a Denver Instruments M-220D with 0.01 milligram place accuracy prior to the experiments. The measured sample mass of the GaN nanowires plus amorphous GaN matrix was 0.0374 grams. The GaN sample was mounted behind a thin ~0.25 mm mica sheet on a p-type silicon substrate (with native oxide) as shown in Figure 4b. The SEETF beam path passed through a 0.075 mm zirconium foil vacuum exit window, a 0.27 mm four-quadrant plastic scintillator plate used to continuously monitor the incoming ^{78}Kr beam current, a 432 mm air gap, and the 0.25 mm mica restraining sheet. Beam energy calculations performed using the Stopping Range of Ions in Matter (SRIM)[12] Monte Carlo code indicated losses of 2.9, 2.7, 4.6 and 5.4 MeV/nucleon, in the zirconium foil vacuum exit window, the four-quadrant plastic scintillator plate, the air gap, and the mica restraining sheet. This resulted in an on-target beam energy of 124.8 MeV/nucleon. The GaN nanomaterial sample was exposed to a uniform 1 x 2 cm^2 beam**Error! Bookmark not defined.** centered on the ~ 0.16 cm^2 sample by laser alignment. The highest radiation dose was 6.4×10^5 particles per second (pps) ^{78}Kr irradiation, for 900 seconds. This resulted in cumulative energy deposition in the GaN sample of ~ 355 Gray (1Gray = 1 Joule/kg).

Seven individual GaN nanowires have been examined to date using HRTEM. Five have shown no visible signs of damage, and their appearance and diffraction patterns are similar to those shown in Figure 3 and reported in Reference (10). Two GaN nanowires have shown evidence of radiation damage. A wide area TEM image of the radiation damage observed in one GaN nanowire is shown in Figure 4(a). A cross section investigation across the nanowire diameter indicated the following. Plumes were always shown to be amorphous with strong concentric rings in their Fast

Fourier Transform (FFT) diffraction patterns, as shown in Figure 4(b). The highly crystalline wurtzite core showed no indication of damage, as shown in Figure 4(c). Gouges on lower side of the nanowire shown in Figure 4(a) did *not* show strong concentric rings, Figure 4(d), and may have been present in the pre-irradiation GaN nanowire.

(3) Pre- and Post Irradiation Characterization of Carbon Nanomaterials

Carbon nanomaterials, including single and multi wall carbon nanotubes and electrospun carbon nanofibers, with well-graphitized vapor grown carbon fibers as controls, were irradiated using a primary beam of Krypton having mass number 86 (^{86}Kr) at 142A MeV. All of these nanomaterials are hollow core graphitic structures with varying wall structures. Single and multi-walled carbon nanotubes (SWCNTs and MWCNTs) have wall structures that are seamless cylindrical single and multiple layer graphene sheets. These are related to vapor grown carbon fibers that have wall structures of well-aligned graphene platelets. Electrospun carbon nanofibers have wall structures of cross linked polymers that vary depending on the choice of polymer monomer and electrospinning conditions.

The pre and post irradiation condition of the samples was investigated using field emission scanning electron microscopy (FESEM), atomic force microscopy (AFM), micro Raman spectroscopy and transmission electron microscopy (TEM). There were no published guidelines for beam exposure times in carbon nanotubes. Therefore, these experiments were conducted with doses and exposure times comparable to those that can be expected to damage or destroy current state-of-the art silicon circuits, as our planned future application is electronic. Failures under dose in a diverse range of silicon devices and technologies indicate damage thresholds of about 10 Gray (Gy) for un-radiation hardened devices and thresholds of about 1000 Gy for radiation-hardened devices. Almost all devices will fail before doses reach 10^5 Gy. Therefore, 100 Gy, 1,000 Gy and 10,000 Gy were set as the target doses. Corresponding times to achieve these doses were calculated to be 15, 90 and 900 seconds.

Samples were placed in thin ~0.5 mm walled quartz tubes (to minimize secondary ion production) and arranged 6 tubes per row, 2 rows deep in a rectangular holder, and placed directly in front of the beam port. The beam port was a zirconium (Zr) foil exit window followed by a short air gap to the target. The energy loss in the Zr foil and the air gap was estimated using LISE[13] to be about 63.13 MeV and 502.3 MeV respectively. After traversing the first 0.5 mm of quartz (density = 2.65 g/cm^3), the energy of the beam was approximately 11,270 MeV. The irradiated area of each sample was estimated to be 16.92 mm^2. For the carbon nanotube, nanofiber, and graphitic fiber samples, the density was estimated as 2.24 g/cm^3 (graphite). With these assumptions, the ions lost approximately 359.2 MeV in the first row of carbon samples, and 419.7 MeV in the second row.

The vapor grown carbon fibers (VGCFs) showed substantial structural damage after 90 seconds of irradiation with very few intact fibers remaining (Figure 5(a))., and total loss of sample by 900 seconds. The electrospun carbon nanofibers showed progressive damage at all radiation doses, with increasing damage at increasing doses. AFM images, shown in Figure 5(b), indicated the fusing of localized damage spots. A film of SWCNTs (buckypaper) was studied to maximize the possibility of an interaction. Pre and post irradiation AFM did not indicate any obvious surface damage at any radiation dose, as shown in Figure 5(c). The MWNCTs were initially in the form of nanotubes mixed with an amorphous carbon growth matrix. Post irradiation FESEM observations of the MWCNTs became increasingly clear for the 15, 90, and 900 sec samples. The 900 sec result is shown in Figure 4(d). It is possible that the ^{86}Kr irradiation caused disintegration of some of the amorphous carbon matrix, similar to its effect on the VGCFs, leaving the multi-wall carbon nanotubes exposed and intact.

Micro-Raman spectroscopy results for the SWCNTs in1300-1700 cm^{-1} region are shown in Figure 6(a). The signature tangential mode double peak[14] for metallic and semiconducting single walled nanotubes was clearly observed at about 1560 / 1590 cm^{-1} for all irradiation times. The planar graphite peak at about 1580 cm^{-1} was not observed for any sample. The Raman spectra were fitted using deconvolution with Lorentz amplitudes. A roughly 10% increase in the full width half maximum (FWHM), from about 18 to 20 cm^{-1}, was observed for both tangential mode peaks progressively with irradiation time, along with a slight shift to lower cm^{-1}.

High resolution TEM images of individual MWCNTs indicated intact wall structures, as shown in Figure 7(a). The Raman signature of MWCNTs is only slightly different from the 1580 cm-1 planar graphite signature and could not be distinguished due to the presence of the amorphous carbon matrix.

Discussion

a. *Nanocircuit Performance*

A GaN nanowire-based field effect transistor circuit has shown normal real-time operation during irradiation by ^{78}Kr heavy ions, under high bias conditions. Normal post-irradiation electronic behavior under high bias conditions was also observed. These results demonstrate the potential that nanowire-based devices may have to function well in radiation environments.

b. *Nanomaterials Performance – Gallium Nitride Nanowires*

The stable performance of the GaN FET nanocircuit is determined by both the nanowire properties and the nanocircuit architecture. The observed radiation response of the two-phase coaxial GaN nanowire structure may be summarized as follows. The majority of the GaN nanowires examined to date did not show strong evidence of radiation coupling; however, these investigations are continuing. Two GaN nanowires did shown evidence of radiation damage. The heavy ion interaction could have resulted in the creation of plumes of amorphous material along the coaxial zinc blende outer layer. The GaN wurtzite core has not shown any evidence of substantial departure from crystallinity to date. All plumes investigated to date have shown strong concentric ring patterns indicating amorphous material in corresponding FFTs. This was also true for outer layers adjacent to the plumes. However, FFTs of the gouges and their adjacent areas have indicated largely crystalline material. Gouges have sometimes also been observed in un-irradiated GaN nanowires. This suggests that heavy ion radiation may be coupling to previously damaged GaN nanowires via defect sites.

c. Nanomaterials Performance – Single and Multi-Walled Carbon Nanotubes

Morphological and molecular bonding characterizations of single wall buckypaper CNT films and multi-wall CNT samples revealed little evidence of damage for 15, 90 and 900 second radiation doses by ^{86}Kr ions at 142 A MeV/nucleon. By comparison, vapor grown carbon fibers showed substantial damage after 90 seconds of heavy ion irradiation and total breakdown by 900 seconds. Electrospun carbon nanofibers showed damage at all radiation doses, with increasing damage at increasing doses. The observed results for vapor grown carbon fibers and electrospun carbon nanofibers are consistent with known displacement and scission radiation mechanisms for graphitic and polymeric materials

The Raman spectra of the SWCNTs showed small increases in FWHM and a faint 1350 cm^{-1} disordered carbon peak at 900 sec. These results are consistent with possible displacement damage. Theoretical investigations of self-healing of displacement damage in carbon nanotubes resulting in compressive stress (Figure 6(b)) have been reported[15] and would be consistent with the observed slight shifts in tangential mode peak locations.

HRTEM investigations of the MWCNTs have not shown any evidence to date for displacement damage resulting in the formation of interstitial planes, of the type shown in Figure 7(b). Such interstitial planes have been observed in MWCNTs in response to electron irradiation[16].

In summary, the performance of nanowire and nanotube materials and actively running nanocircuits during heavy ion irradiation indicates good radiation resiliency compared with control experiments. Some damaging radiation interactions have been observed. The nature of the radiation coupling and the potential of self-healing mechanisms in nanoscale systems are under investigation by our group.

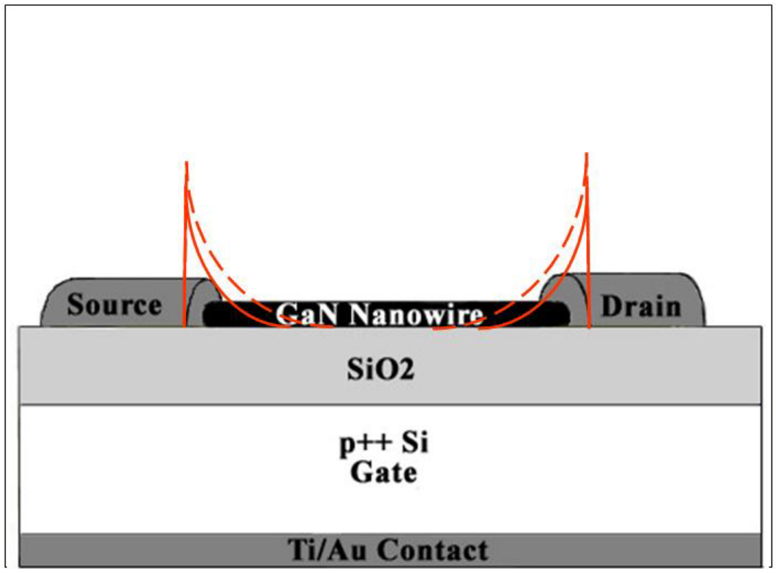

Figure 1. Cross section of GaN nanowire FET. A 150 nm oxide layer is grown on a highly doped p-type silicon wafer functions as the gate. Ti/Au source and drain leads are patterned via electron beam lithography on top of the nanowire, and the Ti/Au gate contact is evaporated on the backside of the wafer forming the global gate. The action of the gate bias VGS is to raise/lower, and thin/thicken the Schottky barrier at the nanowire/nanotube-metal contact.

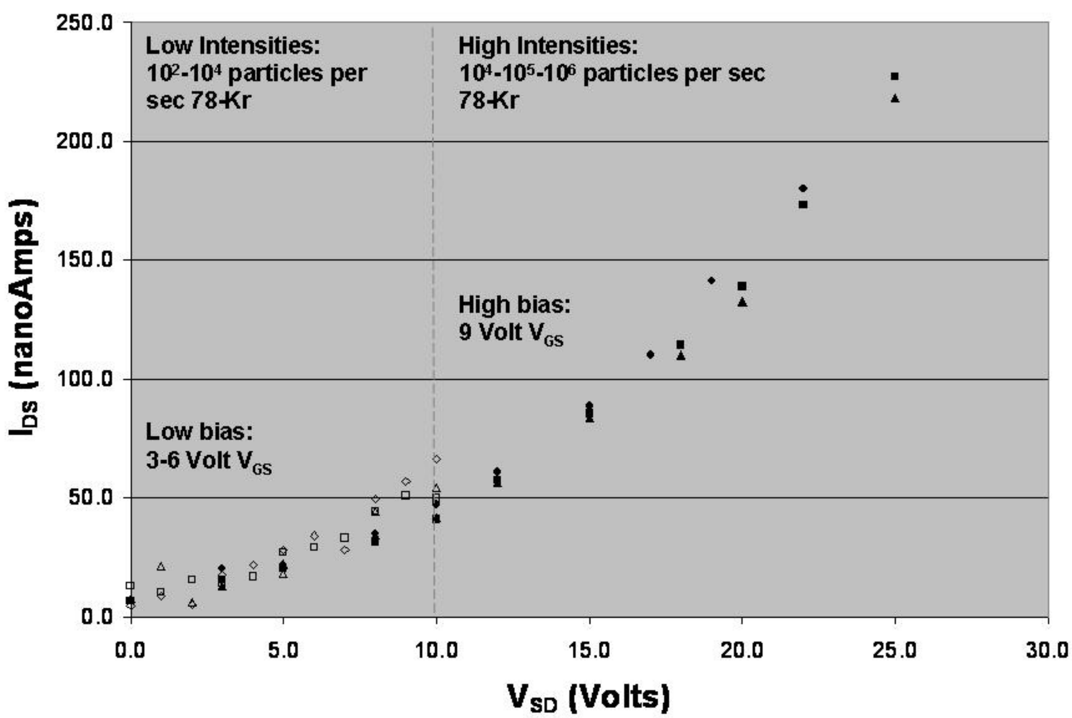

Figure 2. I-V characteristics of actively running GaN NW FET during 78-Kr irradiation.

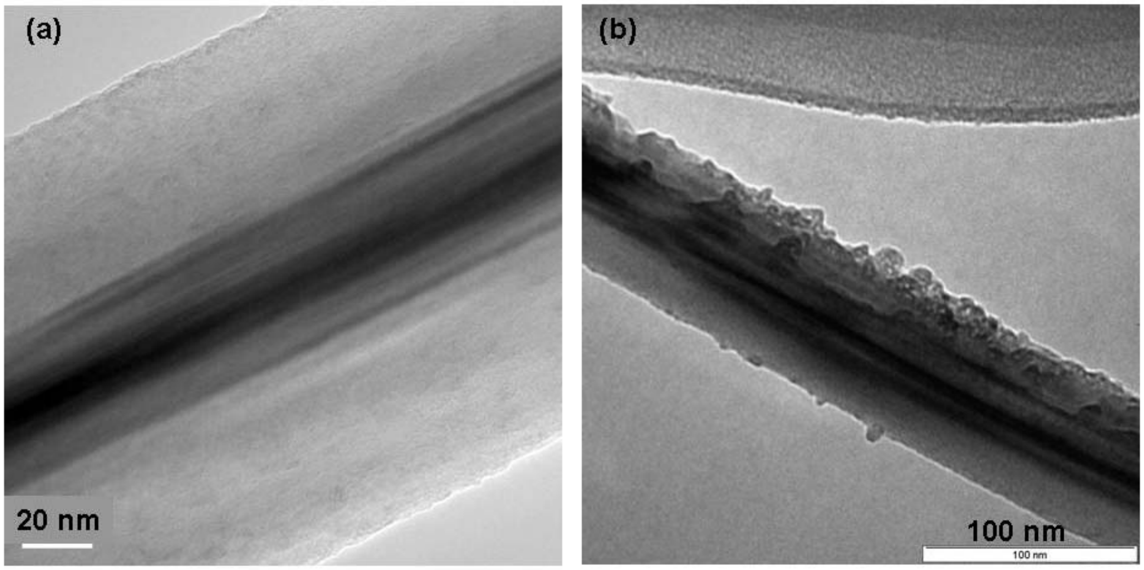

Figure 3. Pre Irradiation GaN nanowires. (a) Most GaN nanowires were highly crystalline with a wurtzite core and coaxial zinc-blende outer layer however (b) defective GaN nanowires were also observed.

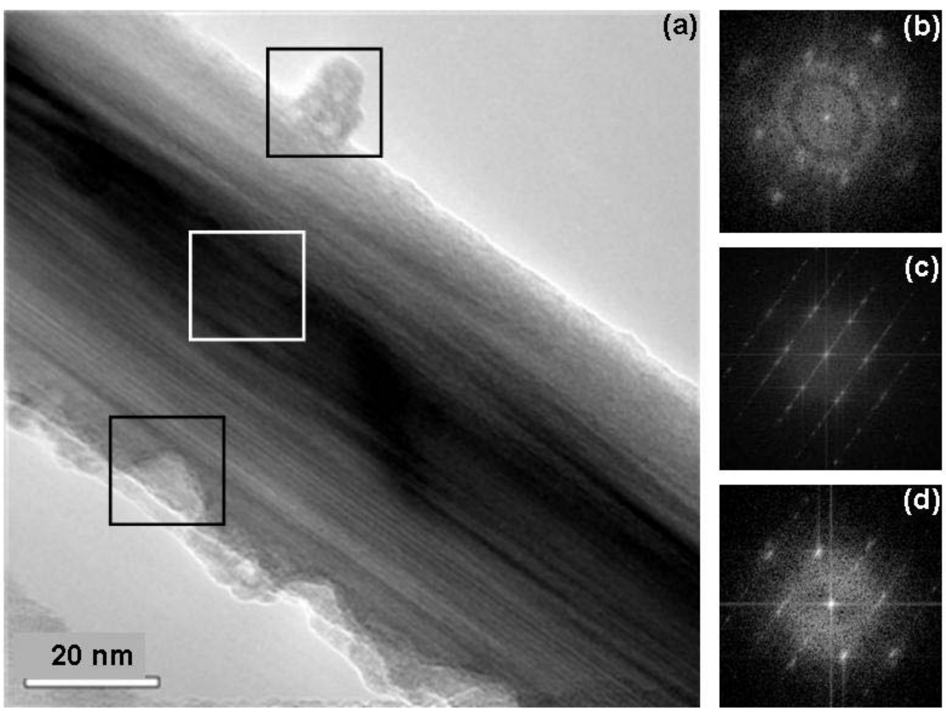

Figure 4. (a) GaN nanowire after 355 Gray 78-Krypton irradiation with corresponding FFTs. (b) Plumes were shown to be amorphous by strong concentric rings in FFT pattern. (c) The highly crystalline wurtzite core showed no indication of damage. (d) Gouges on lower side did not show strong concentric rings and may have been present in the pre-irradiation GaN nanowire.

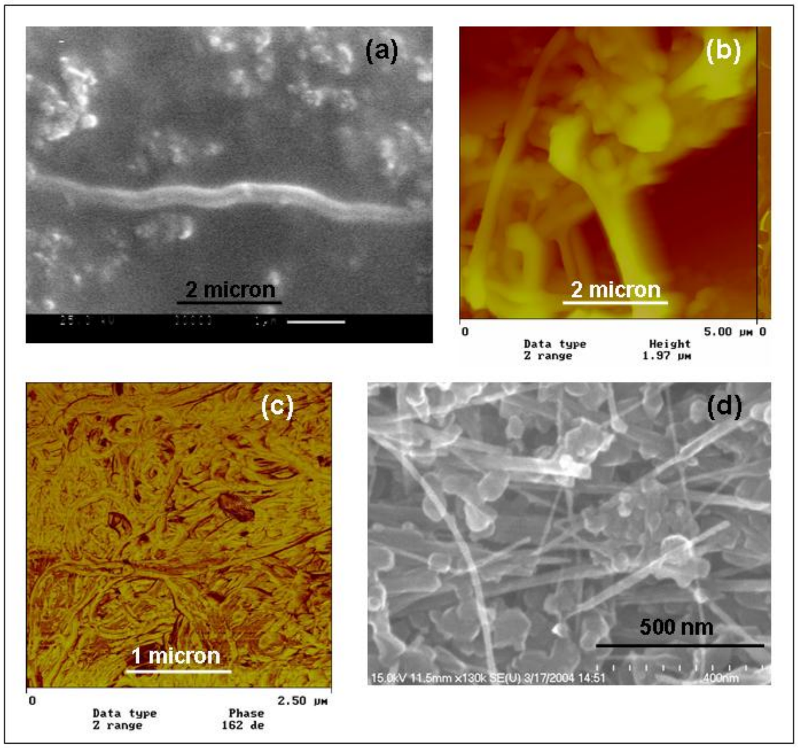

Figure 5. (a) FE SEM of vapor grown carbon fibers, and (b) AFM of electrospun carbon nanofibers indicated substantial structural damage after 900 sec 86-Krypton irradiation. (c) AFM of single walled carbon nanotubes and (d) FE SEM of multi walled carbon nanotubes did not show any obvious structural damage.

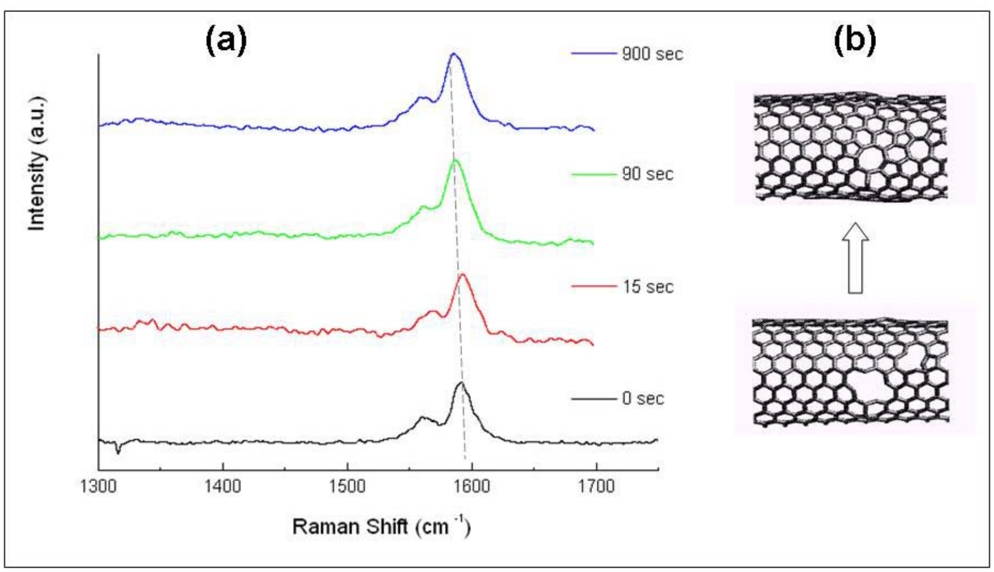

Figure 6. (a) Micro Raman spectroscopy of the tangential modes indicated that the carbon nanotube cylinder was intact for all doses. A slight shift may be due to compressive stress caused by self-healing of displacement damage, as shown in (b) (Figure 6(b) after Reference15).

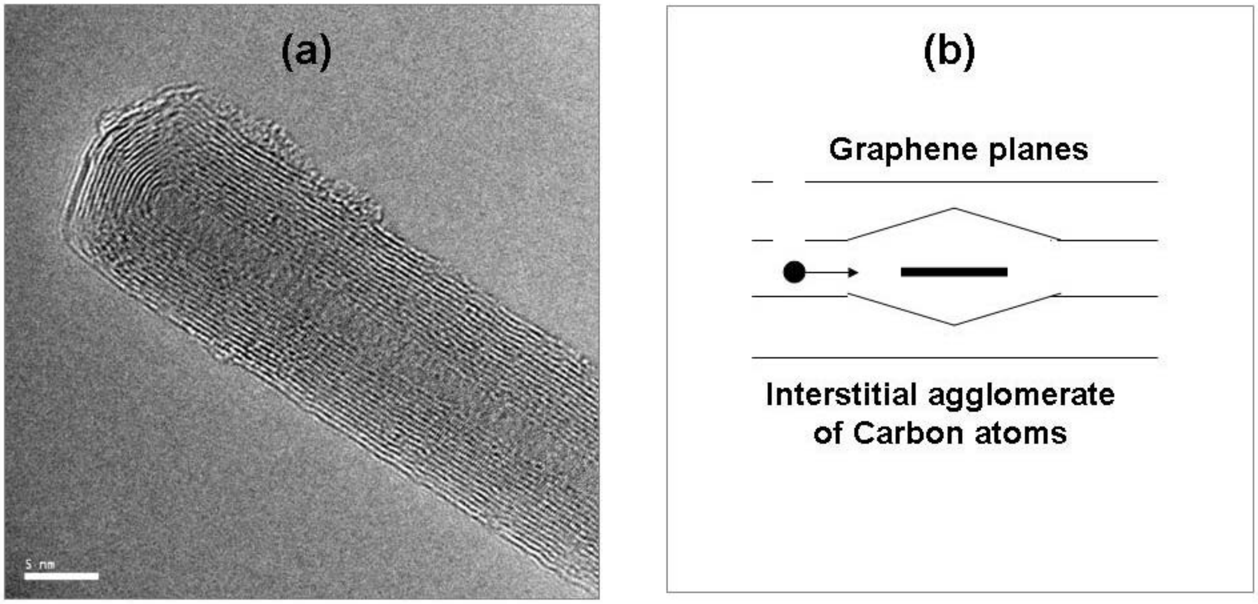

Figure 7. (a) HRTEM image of multi-walled carbon nanotube after 900 sec 86-Krypton irradiation. No displacement damage of the type shown in (b), was observed (Figure 7(b) after Reference 16).

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