Highly conductive ion tracks in tetrahedral amorphous carbon by irradiation with 30 MeV $C_{60}$ projectiles

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Abstract. Electrically conducting ion tracks are produced when high-energy heavy ions pass through a layer of tetrahedral amorphous carbon (ta-C). The tracks are embedded in the insulating ta-C matrix and have a diameter of about 8 nm. Earlier studies showed that the electrical currents through individual tracks produced with Au and U projectiles exhibit rather large track-to-track fluctuations. In striking contrast, 30 MeV $C_{60}$ cluster ions are shown to generate conducting tracks of very narrow conductivity distributions. Their current-versus-voltage curves are linear at room temperature. We also investigated ta-C films doped with B, N, Cu and Fe at a concentration of 1 or 2 at.%. In particular, Cu- and Fe-doped samples show increased ion track conductivity.

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1. Introduction

When high-energy heavy ions (e.g. uranium of 1 GeV) pass through matter, they can induce severe modifications in a very narrow region around each individual trajectory (see e.g. Toulemonde et al 2004, Spohr 2011). Due to the extremely high local energy deposition, the structure of the material may be completely transformed producing a cylindrical track characterized, for example, by an amorphous instead of crystalline phase. Track diameters are of the order of 10 nm; details of size and damage morphology depend on specific properties of the material as well as on various projectile parameters such as the ion mass and velocity. The track length can be varied from a few nanometers up to several micrometers by choosing the appropriate beam energy and/or layer thickness.

Given their small size and long extension, ion tracks represent one-dimensional (1D) nanostructures. At present, there are essentially two ways to apply them in nanotechnology. The first method is based on chemical etching, where each track is converted into an open channel by preferential chemical attack. By exposing, for example, irradiated polymer foils to a suitable etchant, porous membranes are produced containing parallel oriented cylindrical highly monodisperse channels. Such membranes provide a successful route for growing metallic or semiconducting nanowires of well-defined diameter and length by filling material into track-etched channels (see e.g. Piraux et al 1994, Chen et al 2004, Maurer et al 2007).

The second method is applied here and uses ion tracks directly without chemical etching. Tracks consisting of material transformed from the crystalline into the amorphous state are rarely suitable for direct application. In tetrahedral amorphous carbon (ta-C), however, the track not only consists of structurally changed material but is also converted from insulating, diamond-like (sp$^3$-coordinated) carbon into conducting, graphite-like (sp$^2$-coordinated) carbon (Waiblinger et al 1999, Krauser et al 2003, Zollondz et al 2006). The conductivity is sufficiently high to regard the tracks as conducting nanowires embedded in the insulating ta-C matrix. Ion tracks are thus interesting candidates to be used as nanofilaments in more complex electronic devices. Some possible design structures are proposed by Weidinger (2004) and Krauser et al (2008). Recently, a first simple electronic device was fabricated with individual conducting tracks in ta-C in combination with aligned hole electrodes produced by the same ion projectile in a thin polymer resist spin-coated onto the ta-C layer (Gehrke et al 2010).
Figure 1. Schematic illustration of the track formation process in ta-C (a–d). The heavy ion heats the predominantly sp\(^3\)-bonded material to a high temperature. After cooling down, a less dense and sp\(^2\)-bonded material is formed in a narrow cylinder around the ion trajectory (adapted from Schwen 2007).

For application, it is important that the track conductivity is reliably high and does not fluctuate from track to track. In earlier experiments (Krauser et al 2003, Zollondz et al 2003, Nix et al 2007), we found that heavy ions at the upper end of the periodic table (Au or U ions) are necessary in order to get well-conducting tracks. But even for tracks produced with the heaviest monoatomic projectiles (\(^{238}\text{U}\)) at the Bragg maximum of the electronic energy loss (\(dE/dx \sim 40\,\text{keV nm}^{-1}\)), the conductivity data of a given sample showed a rather broad distribution. The scattering is ascribed to the incomplete sp\(^3\)/sp\(^2\) conversion along the ion trajectory representing weak links.

In this paper, we demonstrate that the problem can be overcome by the bombardment of the ta-C film with C\(_{60}\) ions. Such cluster projectiles provide a very high electronic energy loss of \(dE/dx \sim 72\,\text{keV nm}^{-1}\) for 30 MeV C\(_{60}\) ions and produce highly conductive tracks with a remarkable narrow current distribution, representing continuous electric filaments throughout the ta-C film.

A scheme of the track formation process is shown in figure 1. When an energetic heavy projectile (here C\(_{60}\) at 30 MeV) passes through the ta-C layer, it is slowed down by ionization and electronic excitation processes, resulting in an energy transfer of about 72 keV nm\(^{-1}\) along the ion path (figures 1(a) and (b)). This large energy deposition locally heats up the material to several thousands of kelvins for a very short time (figure 1(c)). During subsequent cooling, the formerly sp\(^3\)-bonded carbon atoms rearrange in an sp\(^2\)-bonded structure, yielding conducting cylindrical channels. Due to the lower density of graphite-like carbon compared with diamond-like carbon, the track volume expands, leading to compressive stress in the surrounding matrix. Close to the surface this pressure can be compensated for by an outflow of material forming nanometric hillocks (figure 1(d)).

2. Experimental

The ta-C films were produced at the University of Göttingen by means of mass-selected ion beam deposition (MSIBD) on n-Si substrates with very low resistivity (0.005 \(\Omega\) cm). To produce a 100 nm thick ta-C layer a pre-cleaned and oxide-free Si surface is exposed to 100 eV \(^{12}\text{C}^+\) ions up to a typical charge deposition of 0.4 C. Given by the size of the C beam, the resulting film has a diameter of \(\sim 13\) mm. The process is performed under UHV conditions (10\(^{-5}\) Pa), and due to mass separation, a pure and hydrogen-free film is formed (Hofsäss et al 1993). In addition

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to pure ta-C films, some ta-C layers were doped with Fe, Cu, N (concentration 1 at.%) or B (2 at.%). Doping was achieved by adding the specific element to the ion source and switching the mass separation magnet periodically between $^{12}$C and the dopant ion. Film growth and dopant concentration were controlled via the deposited charge onto the substrate.

Immediately after deposition, the fraction of the sp$^3$-bonded carbon atoms in the films was determined by in situ x-ray photoelectron spectroscopy using Mg K$_\alpha$ radiation (1253.6 eV). The position of the plasmon loss peak with respect to the C 1 s peak maximum was determined and evaluated as described by Reinke et al (2004). For all samples, the plasmon loss energy exceeded 31 eV, indicating that the fraction of sp$^3$-bonded carbon atoms is about 80% or more. For the ta-C:Fe sample, the possible formation of Fe clusters was controlled by transmission electron microscopy (TEM). No such clusters could be found. Previous TEM and x-ray diffraction analyses of Cu-doped ta-C indicate the presence of Cu clusters with size starting from 3 nm diameter for Cu concentrations above 5 at.%. For lower concentrations, the clusters are too small to determine their size with these methods (Gerhards et al 2003).

The irradiation with 30 MeV C$_{60}^{2+}$ ions was performed at the 10 MV tandem accelerator of the Institut de Physique Nucleaire d’Orsay (for details of the C$_{60}$ beam, see Della-Negra et al 2011). To avoid the overlap of neighboring tracks, the applied fluence was limited to about $1 \times 10^9$ ions cm$^{-2}$. When penetrating the sample, C$_{60}$ clusters eventually break apart into smaller C$_n$ fragments. The correlation length in which the C atoms travel together as a cluster is about 100 nm (Dunlop et al 1997). This length is sufficiently long to generate a track in the 100 nm thick ta-C layers. The C$_n$ fragments further disintegrate and eventually stop inside the Si substrate.

C$_{60}$ clusters of 30 MeV lose their energy mainly through electronic excitation and ionization processes, while elastic collisions with the target atoms are predominant near the stopping region. As long as the atomic constituents of the cluster are travelling closely together, the energy loss of the cluster is supposed to be equivalent to the sum of the $dE/dx$ of the constituent individual carbon ions of the same velocity (Baudin et al 1994). According to this rule, the $dE/dx$ of 30 MeV C$_{60}$ ions in ta-C is about 72 keV nm$^{-1}$, which is almost twice the value of 1 GeV U ions.

For comparison, data from 1 GeV Au and U irradiations are included in this paper. The irradiations with Au and U ions were performed at UNILAC of the GSI Helmholtz Centre in Darmstadt. The initial beam energy of 11.4 MeV u$^{-1}$ was reduced by mounting a 50 $\mu$m thick Al foil in front of the samples. The Al foil acts as a degrader and shifts the energy loss of the ions to the Bragg maximum. Moreover, when passing through the degrader, the ions strip off electrons because their charge state as delivered by the accelerator (e.g. Au$^{24+}$ and U$^{28+}$) is lower than the equilibrium charge state of ions at the given velocity. The stripping is a statistical process and the charge state of the projectiles is characterized by a charge state distribution (here approximately $40^+ \pm 3^+$). The range of the Au and U projectiles is several tens of micrometers; the beam thus passes completely through the ta-C layer and stops far inside the Si backing. All irradiations (Au, U and C$_{60}$) were carried out at room temperature and under normal beam incidence.

The conductivity of individual ion tracks was analyzed by atomic force microscope (AFM) measurements at the Hochschule Harz in Wernigerode using an XE-100 AFM (PSIA Corp.). To record $I$–$V$ curves of the individual ion tracks, voltage sweeps were performed while the conductive AFM tip (coated with Cr/Pt) was positioned at the ion impact site (Krauser et al 2008).
Figure 2. AFM images of the topography (left) and the current (right) of a 100 nm thick ta-C film irradiated with 30 MeV C\textsubscript{60} ions. The current was measured between the conducting AFM tip and the Si substrate with an applied voltage of 0.1 V. Each impinging cluster produces a hillock (left) and a current peak (right). Nine tracks are seen on this 1 \(\mu\)m x 1 \(\mu\)m area, consistent with the nominal ion fluence of 1 \(\times\) 10\textsuperscript{9} ions cm\textsuperscript{-2}, which corresponds to 10 ions \(\mu\)m\textsuperscript{-2}.

3. Results

Figure 2 shows the simultaneously recorded mapping of topography and current through an undoped ta-C sample after 30 MeV C\textsubscript{60} irradiation. Each C\textsubscript{60} cluster impinging on the ta-C surface produces a clearly visible hillock (left) and an electrically conducting filament through the sample (right). The peak heights of the hillocks and currents are remarkably uniform. The current peaks are flat at the top, indicating that the current is constant during the time the AFM tip is in contact with the track.

A comparison of the hillock height distributions for different projectiles is shown in figure 3. The average height increases from about 0.8 nm for Au (figure 3(a)) to about 1.6 nm for U (figure 3(b)) and 6.5 nm for C\textsubscript{60} beams (figure 3(c)). Furthermore, the C\textsubscript{60} clusters produce hillocks with a size distribution much narrower than that of hillocks produced by monoatomic ions.

Even more significant is the difference regarding the electrical current signal, as shown in figure 4. The C\textsubscript{60} beam produces tracks characterized by a relatively high and uniform current (figure 4(c)), whereas the data from Au (figure 4(a)) and U ions (figure 4(b)) scatter strongly from track to track and have much smaller values.

Figure 5 shows typical current–voltage (I–V) curves of single-ion tracks in undoped and doped ta-C films. The upper graph (figure 5(a)) displays the data obtained with monoatomic ions (Au or U). There the track currents for the undoped and B- and N-doped samples are rather low and show an upward bending at higher voltages. In the metal-doped films, the currents are relatively high and the I–V curves are linear in the whole voltage range. Thus doping increases the track conductivity, but at the same time in some cases the insulating behavior of the matrix is affected too and the resulting contrast ratio \(I_{\text{track}}/I_{\text{matrix}}\) is reduced, as shown recently by Krauser \textit{et al} (2011).

In contrast to tracks from monoatomic ions (figure 5(a)), all data from the C\textsubscript{60} irradiation (figure 5(b)) display high currents and Ohmic behavior. This finding includes the undoped ta-C
Figure 3. Distribution of hillock heights for 100 nm thick ta-C layers irradiated with Au (a), U (b) and C$_{60}$ (c), respectively. The irradiation parameters (projectile, energy and fluence) are given in the figures. The energy loss $dE/dx$ of these projectiles at the given energy is 33, 42 and 72 keV nm$^{-1}$ for Au, U and C$_{60}$, respectively.

film, for which the current measurements of the Au and U tracks yielded very low values. The Fe-doped sample again shows the highest currents, although the increase of current compared with Au irradiation is only by a factor of 2.5. In all other cases, a much more pronounced increase was achieved with C$_{60}$ irradiation.
4. Discussion

At ambient pressure, the thermodynamically stable phase of carbon is graphite, whereas tetrahedral (diamond-like) ta-C is metastable and is formed only because of the high pressure involved in the fabrication process. High-energy heavy ions passing through ta-C deposit within a very short time a huge amount of energy into a very small volume. Under these extreme conditions, the material along the ion trajectory melts (Toulemonde et al 2000) and expands.
Figure 5. Typical $I–V$ curves of single-ion tracks in undoped and doped ta-C films after irradiation with monoatomic heavy ions (a) and with $C_{60}$ ions (b). In (a), the ions were U (1 GeV) for ta-C:Cu and Au (1 GeV) for all others (Krauser et al 2011).

into the surrounding matrix. As a result, hillocks a few nanometers in height are formed on the film surface and new bonds ($sp^2$ instead of $sp^3$) are established (see figure 1).

The conversion from $sp^3$ into $sp^2$ along the track is a statistical process and depends on the local structure of the original ta-C matrix at a given site, on the distance of the site from the ion trajectory and, of course, on the total energy transferred by the ion. In order to obtain a thoroughgoing conducting filament, the track should not be interrupted; that is, on the atomic scale, at least one conducting bond must exist all along the track. Given by the high conductivity of the $C_{60}$ tracks, we assume that this very stringent condition is fulfilled for the cluster beam, but not in all cases for monoatomic atoms.

4.1. Percolation model

An uninterrupted $sp^2$ string is formed when the probability of $sp^2$ bonds exceeds the percolation threshold, which is 0.39 for the diamond structure in 3D (van der Marck 1998). Here, the bond

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Figure 6. Mean values of the hillock height and electrical current (bias voltage = 0.3 V) of ion tracks in undoped ta-C films. The error bars correspond to the width of the distributions (full-width at half-maximum of a fitted normal distribution) shown in figures 3 and 4. The energy loss dE/dx of the different ions was calculated with the SRIM-2008 code (Ziegler J 2008). For C₆₀, the energy loss was composed of the energy loss of 60 single carbon atoms.

percolation threshold is used. The site percolation threshold is 0.43. This slight difference is not important in the present discussion, in particular since other effects such as the lateral confinement of the system to a narrow cylinder or deviations from the tetrahedral coordination also influence the threshold parameter. In addition, correlation effects, i.e. simultaneous switching of several bonds, may occur or may be required to obtain conductance.

In this model, a conducting track is formed if the probability to find sp²-coordinated carbon atoms exceeds about 0.4. The actual threshold is probably higher, in particular because the lateral extension of the conversion is restricted to the ion track region and because ring structures or partial ring structures are probably required for conductance. In any case, a substantial increase of sp²-coordinated C atoms is necessary in order to accomplish a thoroughgoing path. The original samples have approximately 80% sp³-coordinated atoms, i.e. the sp² probability is about 0.2. At least a doubling of this probability is required to obtain a conducting track. In TEM investigations, the ion tracks can be observed in plane view geometry as pronounced point-like diffraction patterns due to a significant density contrast between the ion track and the ta-C matrix (Schwen 2007). This fact and also molecular dynamics calculations indicate that the actual sp² fraction in the tracks exceeds 50%.

4.2. Dependence on the ion species

Both the hillock height and the track current depend strongly on the ions used for the irradiation. Figure 6 summarizes the experimental results for the different projectiles and sets them in relation to the specific energy loss dE/dx of the ions. The increase in the hillock heights and currents with increasing dE/dx is obvious. A linear fit to the data indicates a threshold of about 30 keV nm⁻¹ for this series of ta-C films. We would like to note that a linear extrapolation of the current data (here measured at a low applied voltage of 0.3 V) does not necessarily yield
Table 1. Conductivity results deduced from AFM measurements for different doped samples after 30 MeV $C_{60}$ irradiation. Conductivities are calculated assuming a conducting cylinder of 8 nm diameter and 100 nm length (thickness of the ta-C layers). The uncertainties represent the standard deviation of the arithmetic mean values.

| ta-C doped with | Track current $I_{\text{track}}$ (nA) at 0.1 V | Track conductivity $\sigma_{\text{track}}$ (S m$^{-1}$) |
|----------------|---------------------------------------------|-----------------------------------------------|
| Undoped        | $4.8 \pm 0.25$                              | $95.5 \pm 5.0$                                |
| N (1 at.% )    | $12.9 \pm 0.75$                             | $243 \pm 15$                                 |
| B (2 at.% )    | $9.9 \pm 0.23$                              | $197 \pm 4.6$                                |
| Fe (1 at.% )   | $53 \pm 2.2$                                | $1054 \pm 44$                                |

the real threshold for track formation, because conducting tracks were also observed at a lower $dE/dx$ of 22 keV nm$^{-1}$ using 140 MeV Xe ions (Waiblinger et al. 1999). But in this experiment a bias voltage of 7 V was applied and a rather low current was measured. Thus, track formation is possible with Xe ions but the track conductivity is rather low.

We suppose that the track currents depend also on the $sp^2/sp^3$ ratio in the pristine ta-C films and that this ratio varies slightly from sample to sample, in particular if the samples are prepared by different methods. The higher the $sp^2$ content of the original ta-C layer, the easier the percolation threshold is reached, yielding to higher track currents (Zollondz et al. 2006). We also found that the initial charge state of the impinging ions plays a role. Higher charge states are more likely to produce well-conducting tracks. At present, no systematic study of the latter effect exists.

The dependence of the hillock height on the impacting ion species, including $C_{60}$, was studied by El-Said (2009) for yttrium iron garnet. There also an approximately linear dependence of the hillock height on $dE/dx$ was found. However, quantitatively there are significant differences compared with our results: in yttrium iron garnet the hillocks produced with $C_{60}$ ions have a height of about 12 nm, which is approximately twice the value found for ta-C and the threshold for track formation is only 5 keV nm$^{-1}$. Tracks in yttrium iron garnet are apparently more easily formed than in ta-C.

4.3. Doping dependence

The results of AFM measurements of undoped and doped ta-C layers irradiated with $C_{60}$ ions are summarized in table 1. The current $I_{\text{track}}$ is the average current through single-ion tracks recorded at a bias voltage of 0.1 V and extracted from AFM current mappings with at least 100 individual tracks. The given uncertainties correspond to the standard deviation of the arithmetic mean values. The track conductivity $\sigma_{\text{track}}$ is calculated assuming a track diameter of 8 nm and a track length of 100 nm (corresponding to the film thickness). The data clearly confirm the trend of earlier measurements on doped films after irradiation with monoatomic ions (Krauser et al. 2011). Slight doping with 1 at.% N or 2 at.% B leads to a moderate metal doping (here Fe) to a strong increase in the track current. In the present case, the enhancement factors are about 2.7 for N, 2.1 for B and 11 for Fe doping. The dopants apparently favor the $sp^2$ bond formation, either already during sample preparation or later in the hot phase of the track formation process. Nevertheless, doping of ta-C with N or B can lead to ambivalent results as an enhanced $sp^2$
content of the pristine material increases its conductivity and hence the current $I_{\text{matrix}}$ through the film as shown by Ronning et al (1995). In consequence, this can lead to an undesired lowering of the contrast ratio $I_{\text{track}}/I_{\text{matrix}}$. To avoid this drawback, doping concentration of N or B should not exceed 1 at.%. 

The catalytic activity of Fe atoms in the ta-C matrix may enhance track conductivity by supporting the conversion from diamond-like sp$^3$ into graphite-like sp$^2$ carbon during the transient liquid phase, leading to the high track currents. We assume that during the growth of the ta-C film, the incorporated metallic impurities are uniformly distributed and thus electrically ineffective so that here the resistivity of the unirradiated film is not influenced. As already mentioned above, Cu concentrations in ta-C of a few at.% can lead to the formation of metallic clusters as found by Gerhards et al (2003). It is obvious that potential metallic cluster formation has to be avoided to preserve the high resistivity of the unirradiated parts of the film.

### 4.4. Comparison with the literature data

Assuming homogeneous conduction within a cylinder of 8 nm diameter, one obtains conductivities of the order of 100–1000 S m$^{-1}$ (see table 1). Bulk graphite has a conductivity of $727 \times 10^2$ S m$^{-1}$ (Weast 1980), which is two to three orders of magnitude higher than the track conductivity observed here. Thin wires have a reduced conductivity due to electron scattering on the wire boundaries and due to boundary roughness. An important parameter in this respect is the ratio of the wire diameter, here $d = 8$ nm, to the charge carrier mean free path $\lambda$. For graphite, reported values are $\lambda = 235$ nm (Kinchin 1953) and $\lambda \approx 200$ nm (García et al 2008). The very small value $d/\lambda \approx 0.034–0.04$ gives a reduction from bulk to wire conductance by one to two orders of magnitude according to Dimmich and Warkusz (1986). Thus, the conductivities in the ion tracks for C$_{60}$ irradiation are only about one to two orders of magnitude smaller than what is expected for thin graphite wires.

Moreover, our samples are amorphous and the electronic transport is most likely dominated by hopping processes, in contrast to the metallic conduction in crystalline graphite. In a study of sputter-deposited amorphous carbon films (Dasgupta et al 1991), the highest conductivities measured at room temperature were about 0.1 S m$^{-1}$. These values are several orders of magnitude smaller than those obtained here, although a large sp$^2$ content is reported for these films. The authors suggest that the sp$^2$ atoms are grouped in islands, which are separated by non-conducting sp$^3$ regions, leading to rather low overall conductance. In a survey on ion beam sputtered amorphous carbon films (Dawson et al 1995), conductivities between 9 and 840 S m$^{-1}$ are reported, comparable to what we found here in the tracks. In another study, amorphous carbon–nickel composite films (10% Ni) were investigated (Bhaattacharyya et al 2006). These films have conductivities of about 800 S m$^{-1}$ at room temperature comparable to our values for doped films. Thus, the conductivities in the tracks are comparable to or higher than those found in thin amorphous carbon films. In all these materials, the sp$^2$/sp$^3$ ratio plays a crucial role and the clustering of sp$^2$ bonds is important.

### 5. Conclusion

A significant improvement of the electrical conductivity of ion tracks in ta-C was achieved by using C$_{60}$ clusters instead of monoatomic heavy ions for the irradiation. Track-to-track current fluctuations are minimized and all tracks exhibit almost identical high conductivity.
Conductivities measured for tracks from \( \text{C}_{60} \) clusters are about 1–10% of the values predicted for metallic graphite wires a few nanometers thick. Stable conductivity is certainly a substantial advantage for systematic studies, e.g. for investigations of the conductivity mechanism, but also for possible applications of conducting tracks in nanoscale electronic devices.

The improvement achieved with \( \text{C}_{60} \) beams is attributed to the higher-energy loss of the cluster ions compared with that of monoatomic ions. The track produced by a \( \text{C}_{60} \) cluster seems to be a continuous string of conducting \( \text{sp}^2 \) bonds. In contrast, tracks of monoatomic ions barely reach the percolation threshold and due to statistical fluctuations possibly contain electric interruptions with an immense effect on the conductivity.

As already shown by Krauser et al. (2011), the conductivity of the ion tracks can also be improved by doping the ta-C film, in particular with metal atoms such as Cu or Fe. The effect on the conductivity can, however, be countervailed by a decrease in the insulating capability of the ta-C matrix. Similarly, a larger \( \text{sp}^2 \) content in the pristine ta-C matrix improves the conductivity of the ion tracks but degrades the insulating behavior of the matrix.

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