CHEMISTRY OF
MATERIALS
Article

Chem. Mater. **2010,** *22,* 5625–5629 5625
DOI:10.1021/cm102005m

Preparation and Evaluation of Graphite Oxide Reduced at 220 °C

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Received July 19, 2010. Revised Manuscript Received August 16, 2010

The reduction of graphite oxide (GO) thin films was evaluated at 220 °C using a combination of infrared (FTIR) and X-ray photoemission spectroscopies (XPS). The results were correlated with electrical resistance measurements. The chemical composition of GO was $C_8(OH)_3O_{0.8}$ and reduced to $C_8(OH)_{0.5}O_{0.3}$ after nearly 24 h of low-temperature processing, defined as 220 °C. The sheet resistance of dropcast GO thin films processed at 220 °C in air was 8 $k\Omega\ sq^{-1}$, similar to GO reduced at >800 °C in an inert atmosphere.

1. Introduction

Graphite oxide (GO) is a highly oxidized, planar material comprising 25−33% oxygen intercalated in a disordered cyclohexenyl matrix.[1−4] The presence of oxygen makes GO water-soluble and amenable to wet chemical deposition and processing strategies, but disrupts the extended sp^2 network found in conductive 1D and 2D carbon materials.[1−7] To become a conductive material, GO must be reduced. The excellent thermal, mechanical, and material properties of reduced GO have been used in areas of polymer composites, energy storage, sensing elements, field-effect transistors, and transparent electrodes.[8−11] Sheet resistances $<1\ k\Omega\ sq^{-1}$ with better than 80% optical transmittance have been measured for reduced GO, values approaching those of inorganic transparent conductors.[11]

Chemical and thermal methods have been developed to reduce GO.[2,3,7,11−17] Most chemical methods use strong reductants such as hydrazine or sodium borohydride, although vitamin C has also recently been demonstrated.[2,3,5,6,11−13,16−18] Thermal reduction studies have used temperatures above 800 °C, typically in an inert atmosphere.[11,19] Thermal gravimetric measurements have shown two mass transitions as GO is heated, the first being the loss of interstitial H_2O around 110 °C and the second exothermic mass loss (30% of total mass) occurring at 220 °C.[11,14,15,19−21] Other techniques, such as X-ray diffraction, have confirmed the thermal transformation around 220 °C.[14,15] Reduced GO contains 8−10% oxygen using chemical or thermal methods.[3,5,11−13,15,17,22] Sheet resistances of chemically reduced GO are typically more than an order of magnitude higher than those using thermal reduction, presumably because of the incorporation of impurities in the reduction product.[2,3,11,13] Work in the Bao laboratory suggests that the final structure of the sp^2 framework greatly impacts the material characteristics of reduced GO.[11]

The temperatures used for most thermal GO reduction procedures are above back end of line thermal budgets (350−400 °C for 60 min) used in semiconductor device manufacturing thereby limiting the utilization of GO in certain manufacturing processes.[23] This study focuses on mitigating this limitation using low temperature GO

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reduction (defined here as 220 °C). A combined spectroscopic and electrical conductance approach is used to characterize GO and the reduced product as a function of thermal processing time at 220 °C.

2. Experimental Section

All materials were obtained from commercial sources. GO was prepared from a modified Hummers method using 0.5 g of graphite powder dispersed in 23 mL of H_2SO_4 kept at 0 °C. Next, 0.5 g of $NaNO_3$ and 3 g of $KMnO_4$ were slowly added.[7,24] After the solution was a well-mixed slurry, it was transferred to a 35 °C water bath and stirred for 1 h. The temperature was raised to 90 °C after the addition of 140 mL of H_2O. The solution changed to a light brown color during the dropwise addition of 3 mL of 30% H_2O_2. The warm solution was filtered and washed with one volume of warm 18.2 MΩ cm water and centrifuged at 4000 rpm for 4 min. The filtrate was resuspended and centrifuged. The resuspension/centrifugation process was repeated several times.

GO thin films were dropcast from dilute aqueous solution onto cleaned SiO_2 microscope slides and dried in a vacuum desiccator at room temperature. The films were kept in the desiccator until they were visibly dried (60−90 min) and then transferred to a 110 °C oven for 3 h to remove bulk and interstitial H_2O from the films (annealing).[7,11] XPS showed no detectable change in the film structure after annealing. Samples were covered and placed in a 220 ± 2 °C oven in laboratory air (22% O_2) for a prescribed amount of time. The samples were characterized at room temperature. Frequently, all or part of the reduced films delaminated from the SiO_2 substrate during annealing.

Infrared spectra were acquired from pellets of nominally 1% GO in KBr from dropcast GO that was dried in a vacuum desiccator for 12−36 h. KBr was kept in a desiccator prior to use to minimize adsorbed H_2O from the powder. A commercially available Fourier transform infrared (FTIR) spectrometer was used in transmission mode, and spectra were referenced to a KBr pellet without GO. For consistency and data comparison, data were collected on the same KBr samples after 110 °C and/or 220 °C treatment.

X-ray photoemission spectroscopy (XPS) data were collected on a commercial spectrometer equipped with a high resolution, multichannel-array detector, and using a monochromatic Al Kα (1486.7 eV) source using a 40 eV pass energy. The typical pressure during analysis was 2×10^{-9} Torr. Samples were typically kept under UHV for several hours prior to characterization. An electron gun was used for charge compensation. The energy scale was referenced to the C 1s C−C (C0) peak at 284.6 eV. Instrument-specific relative sensitivity factors were used for quantification. Peaks were fit using a 80:20 Gaussian:Lorentzian peak shape after Shirley background subtraction.

Ultraviolet photoemission spectra (UPS) were acquired using a He I source (21.22 eV), a 5 eV pass energy, using a −5.75 V bias. Work function measurements were calculated from the onset of the vacuum level cutoff. The energy scale (Fermi level) was referenced to clean gold.

Sheet resistance measurements were made by using a 4-point probe arrangement on thermally reduced GO films and are reported as Ohms per square (Ω sq^{-1}). The 4-point probe experiment was calibrated against NIST SRM 2546 (lightly doped Si wafer with known resistance). This data was also checked for any geometric anomalies using the National Bureau of Standards Technical Note 199. Thermal gravimetric measurements were made on 3 cm diameter GO films collected on filter paper that were dried through a vacuum filter in air for 12 h prior to characterization. Mass measurements were made using a commercially available high sensitivity balance after 3 h at 110 and 220 °C.

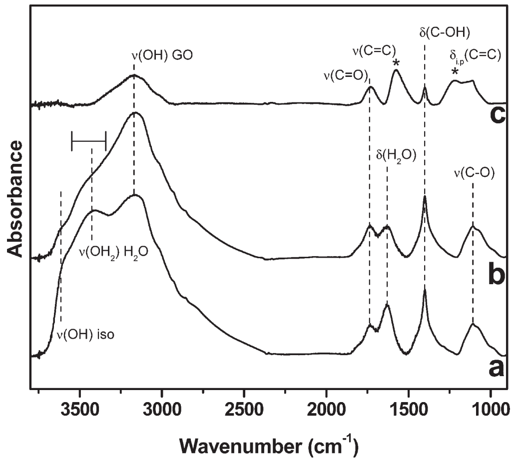

Figure 1. FTIR from KBr pellet of (a) GO, (b) GO annealed at 110 °C for 20 min, (c) GO processed at 220 °C for 45 min. Peaks marked with * are assigned to reduced GO.

3. Results and Discussion

Infrared spectra of GO obtained from KBr are shown in Figure 1. The spectrum of vacuum-dried GO (Figure 1a) shows significant contribution from −OH and C=O functionalities, consistent with previous infrared spectra of GO.[1,25] At least three separate ν(OH) modes can be identified with principal absorptions at 3617, 3418, and 3166 cm^{-1}. These are assigned as isolated hydroxyl groups, intercalated H_2O, and −COH within GO, respectively. These assignments are bolstered by corresponding δ(OH_2) modes at 1630 cm^{-1} for H_2O and an intense, sharp mode at 1402 cm^{-1}, typical for in-plane −COH bends. The mode at 1732 cm^{-1} is assigned to ν(C=O). The peak at 1120 cm^{-1} is consistent with ν(C−O). Studies have shown that GO loses interstitial H_2O around 110 °C.[7,11,20,26] The H_2O modal intensity (3418 and 1630 cm^{-1}) was reduced nearly 50% after 20 min of 110 °C heating (see Figure 1b). Dropcast films lost 66% of their mass after 3 h of 110 °C annealing, although −COH and C=O modes were unaffected. Changes in weight and infrared assignments are shown in Table 1. Films processed at 220 °C became opaque and lost 31% of their remaining mass, consistent with previous gravimetric measurements.[11,26] Thermal processing at 220 °C removed the remaining H_2O modal intensity (Figure 1c). Modes associated with −COH and C=O lost 60 and 30% of their intensity, respectively, after 220 °C thermal processing. Reduction at 220 °C is further evidenced by new modes at 1577 and 1230 cm^{-1}, assigned as aromatic ν(C=C) and in-plane δ(C=C).[1,19,25,27]

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Table 1. Thermogravimetric and FTIR Peak Assignments and Positions (cm^{-1}) for Vacuum-Dried, 110 °C Annealed, and GO Processed at 220°C

	peak position (cm^{-1})^b		
peak assignment	vacuum-dried GO	110 °C^a	220 °C^a
$\nu(OH)_{isolated}$	o	o	n/o
$\nu(OH_2)_{water}$	o	o	n/o
$\nu(OH)_{-COH}$	o	o	3166
$\nu(C{=}O)$	1732	o	o
$\delta(OH_2)$	1630	o	n/o
$\nu(C{=}C)_{aromatic}$	n/o	n/o	1577
$\delta(-COH)$	1402	o	o
$\delta\ (C{=}C)_{in\text{-}plane}$	n/o	n/o	1230
$\nu(C-O)$	o	o	o

[a] Mass change from previous was 65.8 amd 30.9% for 110 and 220 °C, respectively. [b] [ν] stretch, [δ] bend, [o] observed, [n/o] not observed.

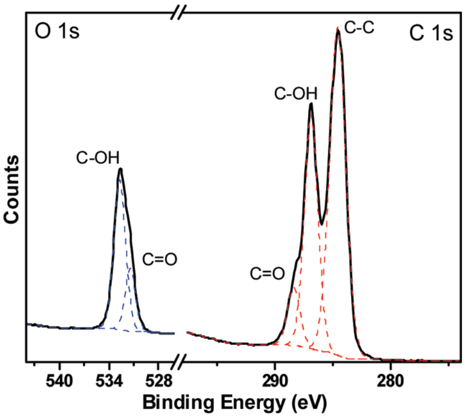

Figure 2. O 1s and C 1s XP spectral regions of GO annealed at 110 °C for 3 h. Dashed lines show component fits in each spectral window.

The full XPS elemental analysis was consistent with the presence of only carbon and oxygen in the graphite oxide. XPS data of the C 1s and O 1s bonding regions of a GO thin film after 3 h of 110 °C annealing is shown in Figure 2. The carbon to oxygen ratio (C:O) of GO annealed at 110 °C was 2.0 using the C 1s and O 1s spectral manifold intensities. Other studies have measured C:O values between 2.0 and 3.75.[1,5,12,13,17] The C 1s region can be deconvoluted into 3 components corresponding to three carbon bonding environments within GO: C−C at 284.6 eV (C0), C−O at 286.7 eV (C1), presumably as C−OH (vide infra), and C═O at 288.0 ± 0.2 eV (C2). The relative peak intensities of the states are qualitatively similar to those previously reported using the Hummers synthetic protocol, showing that nearly 50% of carbon is bonded to oxygen.[3,5,11,12,19,27] Further insight into the carbon−oxygen bonding is garnered from the O 1s peak manifold, also detailed in Figure 2. In a similar manner, the O 1s peak manifold can be fit with two components.[12,27] Here, a peak assigned to O═C is observed at 531.0 eV (O2), and a second more intense component at 532.4 eV (full width half max, fwhm, = 1.7 eV), assigned to the O−C (O1), as C−OH (vide infra).[28]

The chemical composition of GO has been the focus of previous studies, as it is important to understand what chemical functionalities are present in GO to design facile and efficient reduction strategies.[1,5,12,13,17] Several models of GO have been proposed that contain functionalities such as alcohol, ether, ketone, lactol, and acid functional groups interspersed in a cyclohexenyl matrix.[1,4,22,29] The similarity of peak positions in XPS can make assignment of the functional groups in graphite oxide difficult. Here the GO chemical composition was aided using quantitative analysis of carbon and oxygen XPS components and the previously described infrared spectra. One method to determine chemical composition is from the ratio of chemical components (such as C1 for C−O and O1 for O−C).[28] For example, alcohol (−COH) groups would expect to have C1:O1 of 1, whereas an ether (C−O−C) would have a value of 2. A similar analysis using C2:O2 can be made for C═O to distinguish between C═O and COOH. The C1:O1 was measured for several GO films to be 1.2 ± 0.1, similar to −COH or −CH = O. Likewise, the C2:O2 was 1.2 ± 0.1, typical of a ketone (C═O). Infrared spectra show modes are also consistent with −COH and C═O functional groups. Using the C:O and the above component analysis the chemical composition of GO was $C_8(OH)_3O_{0.8}$, similar to the GO composition found by Szabó and others.[1] Structural models of GO containing −COH and C═O have been proposed by Nakajima et al. and Scholz and Boehm.[30,31] In these models GO exists as alternating cyclohexenyl and cyclohexyl rings modified by alcohol and ketone functionality.[30,31] A similar, but more alcohol-rich, model could explain the GO prepared in this study. It can be expected that based on the aggressive oxidation steps required for GO preparation and the variety of modifications from Hummers synthetic routes between research laboratories, that perturbations can alter the final GO product.[1]

XPS data recorded from GO films as a function of processing time at 220 °C are shown in Figure 3. The initial C:O of GO is near 2 and is increased to 3.2 after 4 min of 220 °C processing, although the C 1s spectral envelope is nearly unchanged from annealed GO. Although the C:O increases to 9.8 after 16 min of thermal processing, the most obvious change is the loss of nearly 75% of both C1 and O1 component intensities, most likely as −COH. The formation of new sp^2 C−C bonds is evidenced by a 33% increase in the C0 component. In addition to loss of C1 and O1 intensities, the C1 is shifted 1.1 eV to lower binding energy. The fwhm of the thermally processed O1 component is 1.6 ± 0.1 eV, within uncertainty of the fwhm of nonprocessed GO. An equal shift to higher binding energy is measured for the O1, and is similar to that observed by Yang et al.[12] This is interpreted as reduction of the C−O bond strength. Other components are not affected, indicating the change in the chemical composition associated with the C1 and O1 components do not affect the core electron densities of C═C nor C═O. The component ratio (O1:O2) within the oxygen peak manifold is 3.75 for annealed GO and is

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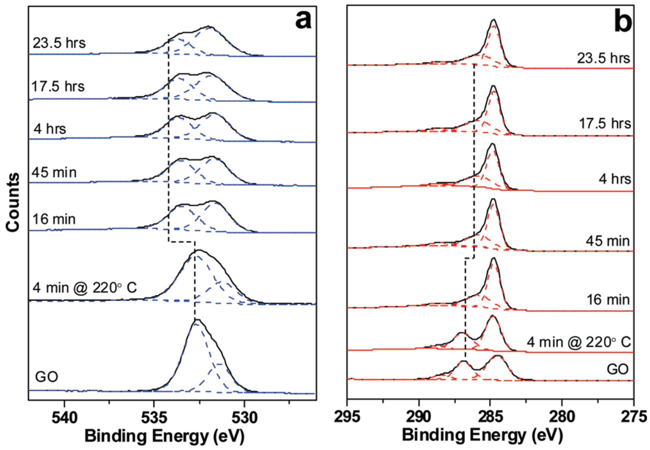

Figure 3. (a) O 1s and (b) C 1s XP spectral regions of GO annealed at 110 °C, and further processed at 220 °C for 4 min, 16 min, 45 min, 4 h, 17.5 h, and 23.5 h. Dashed line shows 1.1 eV shift for C1 and O1 components to lower and higher binding energies, respectively, after 16 min of processing at 220 °C.

Table 2. Component Intensities and Chemical Composition of GO and after Processing Film for 23.5 h at 220°C

	C 1s			O 1s			
	C0 [C−C]	C1 [C−O]	C2 [C=O]	O1 [O−C]	O2 [O=C]	C:O	formula
GO	51.4	39.0	9.6	81	19	2.0	$C_8(OH)_3O_{0.8}$
23.5 h	68.0	25.6	6.3	31	69	10.1	$C_8(OH)_{0.5}O_{0.3}$

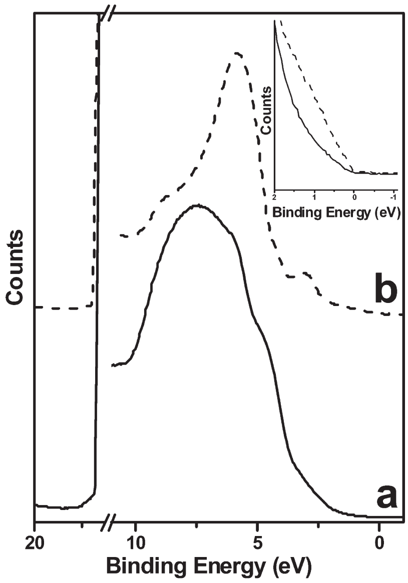

Figure 4. Ultraviolet photoemission spectra (UPS) low binding energy region of (a) GO processed at 220 °C for 23.5 h (solid line) and (b) HOPG (dashed line). Fermi level region shown in inset.

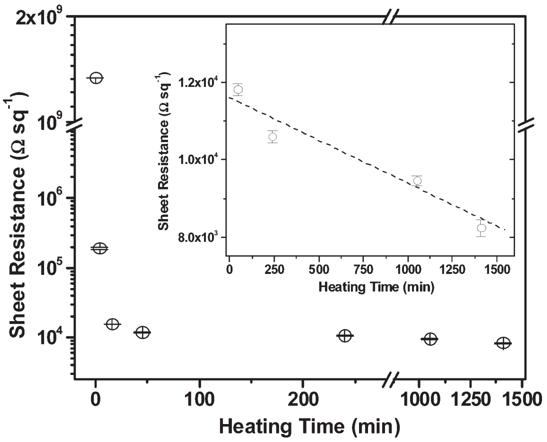

Figure 5. Sheet resistance as a function of GO processing at 220 °C. Inset shows processing times > 16 min. Error bars represent 1σ.

reduced to 0.90 after 16 min of heating, and to 0.48 after 23.5 h. Component intensities are shown in Table 2. XPS and FTIR data both show that low temperature thermal processing results in the loss of −COH and C=O. XPS shows that 75% of −COH and nearly half of C=O is removed. The shift in binding energies in the C1 and O1 components make a definitive assignment of the C−O bonding environment somewhat ambiguous, using the C:O, O1, and O2 component intensities the final reduction product composition is $C_8(OH)_{0.5}O_{0.3}$. Interestingly, the C:O measured with XPS is similar, or larger than those obtained after 24 h of hydrazine reduction or heating GO to 1100 °C under hydrogen.[6,12,13]

Ultraviolet photoemission spectra (UPS) were collected after 4 min of thermal processing, suggesting that film conductance and charging becomes negligible after short processing times. Shown in Figure 4 are ultraviolet photoemission spectra (UPS) of GO processed at 220 °C for 23.5 h and freshly cleaved highly ordered pyrolytic graphite (HOPG), a model 2D aromatic network.[32] Both spectra show density of states at the Fermi level (zero binding energy) associated with hole transport. This density is likely from π−states consistent with an extended aromatic network in both materials.[32−34] Other π-states are observed below 5 eV and σ-states at higher binding energies (see inset of Figure 4). The UPS data of GO processed at 220 °C is similar to that of GO reduced at 1100 °C.[33] Both materials show an onset of the highest occupied molecular orbital (HOMO) at nearly 2.0 eV, although the π-states of thermally processed GO are not as well defined as those observed in HOPG. Low binding energy HOMO onsets are typical of conductive materials.[34] The work function for thermally processed GO is 4.4 eV, identical to that measured for HOPG.

The sheet resistance as a function of thermal processing time at 220 °C is shown in Figure 5. The sheet resistance of annealed GO was greater than 10^9 Ω sq^{-1}.[11] The sheet resistance was 1.90 × 10^5 Ω sq^{-1} after 4 min of 220 °C thermal processing and was reduced to 1.5 × 10^4 Ω sq^{-1} after 16 min of heating. Further 220 °C thermal processing resulted in a slower but monotonic reduction (2.3 Ω sq^{-1}/min) with processing time. The lowest sheet resistance was measured as 8200 ± 200 Ω sq^{-1} after 23.5 h at 220 °C, slightly higher than GO reduced at 1000 °C.[11] Becerril speculated that a modest change in the bonding environment in nonoxygenated carbon had a significant

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impact in the electrical performance of GO reduced at 1000 °C.[11] This is also supported by these data. After 45 min of processing, the C:O is nearly constant with heating time. The C0 intensity is unchanged at 64 ± 3% (1σ) of the total C 1s envelope at > 45 min heating times, although the sheet resistance continues to slightly improve. This is most likely explained by the rearrangement of the sp^2 bonding network, thereby lowering the film sheet resistance with processing time.

Film compositional and electrical measurements show that GO undergoes thermal reduction at 220 °C in ambient laboratory air (22% O_2), conditions more oxidative than those typically used in semiconductor processing. X-ray photoemission, infrared spectra, and sheet resistance each give some insight into the physical process of GO reduction at 220 °C. FTIR and XPS data clearly show thermal processing at 220 °C reduces GO through the loss of oxygen and the formation of new sp^2 C−C bonds as evidenced by the loss of C1 and O1 components and concomitant growth of C0. Careful thermal desorption mass spectroscopic measurements by Jung and others have shown that CO, CO_2, and H_2O are liberated from GO below 220 °C, and likely results in the reduction of the C1 and O1 component intensities here, as well as the loss of OH and C=O modes observed in the FTIR results.[20] The loss of gaseous carbon compounds likely opens voids in the sp^2 matrix which may ultimately limit the sheet resistance of reduced GO compared to 2-dimentional conductive carbon-based materials, such as graphene. New sp^2 C−C bonds are manifested in the conductance with more than a 5 orders of magnitude reduction in the measured sheet resistance. GO reduced at 220 °C contains nearly 10 atomic percent oxygen after reduction, likely as C=O and −COH. The C:O and sheet resistance data both illustrate that modest changes in the aromatic network can provide a large impact to sheet resistance of thermally processed GO films.

Acknowledgment. The author acknowledges Drs. Christina Hacker, Brent Sperling, Roger van Zee, Rebecca Zangmeister, and Gery Stafford for assistance in this investigation. The NIST Office of Microelectronics Programs funded this work.