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COMMUNICATION

Non-destroyable graphene cladding on a range of textile and other fibers and fiber mats

Cite this: DOI: 10.1039/x0xx00000x

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Received 00th January 2014,

Accepted 00th January 2014

DOI: 10.1039/x0xx00000x

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Electrically insulating textile and synthetic fibers were cladded with chemically modified graphene via a three-step technique to induce electrical conductivity. Electrical conductivities of 13 and 4.5 S/m were obtained for aramid and nylon fibers, respectively. The graphene cladding is non-destroyable when washed in detergent or sonicated.

Natural as well as synthetic materials for fabrics and textiles such as cotton, nylon, polyester, and wool are well known for their thermal and electrical insulating properties. However, for applications such as harvesting energy from wearable devices and electromagnetic interference (EMI) shielding effectiveness in the defense industry, good electrical conductivity is required^[1]. Therefore, fabrics used for the defense applications are made of metal wire meshes or metal wire meshes woven along with the textile fibers such as cotton, nylon, polyester, and wool. In some studies, gold and silver nanoparticles have been entrenched inside the microstructure of textile fibers^[2]. There are reports of grafting and polymerization of conducting polymers onto fabric materials to induce electrical conductivity^[3]. In these studies, potential usage of the textile materials in making wearable electrical devices are proposed, and making transistors and capacitors successfully using cellulose/paper-nanoparticles-polymer hybrid fabric have been reported^[4]. Hu et. al. reported coating cellulose microfibrils with carbon nanotubes (CNTs), resulting in high electrical conductivity and forming of a capacitor^[1b] whereas in another report, Alba et. al. applied CNTs on cotton fibers^[5]. However, the CNTs are deposited on cellulose microfibrils and cotton fibers as particles rather than wrapping individual fibers and weak CNT-fiber surface adhesion resulted in non-washable fabric/fiber. Liu et. al. decorated CNTs on chitosan and cotton, added into the electrical properties of these materials while retaining the intrinsic properties of CNTs, however, the resulting material has not shown to be non-destroyable, for example, by washing. And it is not clear if the process is adaptable to other fabric

materials as well^[6]. The inevitable demand for renewable and sustainable energy resources makes such research work potentially important for future applications. However, their complex synthesis procedures, use of expensive materials, and their narrow compatibility with polymer materials is a major hindrance for these materials to be practical. There are existing technologies for converting body heat to electricity^[7], charging devices using the same electricity^[8], to heat or cool our body depending on the season, running wearable devices with our own body energy^[9] and similar other such technologies^[10]. In order to exploit these technologies with fabrics and textiles and to possess good EMI shielding effectiveness without embedding the fabrics with expensive and not-so-wearable gold or silver nanoparticles or copper or nickel wires, there is an immediate demand for an innovation in making the existing fabric and textile materials electrically conductive with an efficient, adaptable, and economical method. An invention possessing the said attributes will make several technologies immediately practicable. It has been envisaged that conducting layers on top of individual fabric fibers will shape the future for textile based wearable electronic devices^[14].

Graphene is a two-dimensional (2D) modern-day marvel material with extra-ordinary properties useful for a wide range of applications and has attracted attention due to its varied properties such as high electrical and thermal conductivity, extraordinary elasticity and stiffness etc.^[11] Addition of graphene can enhance mechanical properties and electrical and thermal conductivities of polymer composites due to its unique nanostructure^[11b]. There are a number of methods for fabricating graphene and chemically modified graphene from different carbon materials including carbide compounds and graphitic derivatives^[11b, 12]. Each of these methods has different advantages, disadvantages and applicability to different fields of sciences and engineering. Preparation of chemically modified graphene, usually referred to as reduced graphene oxide (rGO) from colloidal suspensions is flexible, scalable, and adaptable to a wide variety of applications^[12].

Herein, we propose a facile technique of cladding several textile materials, with rGO. The proposed method is a facile three stage technique which induces high electrical conductivity to the subject materials. The method is shown schematically in Figure 1 which involves three major steps named as build-up, layering, and reduction.

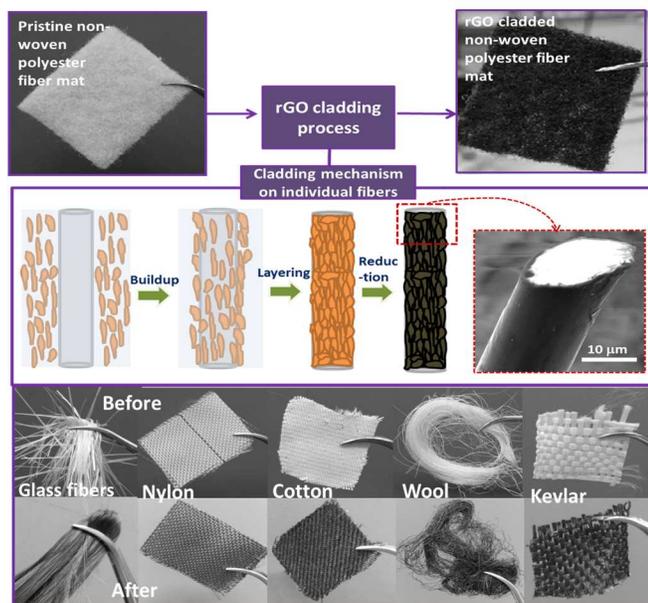


Figure 1: Schematic of the cladding process, mechanism and photographs of cladded materials.

In the build-up stage fibers are dipped in graphene oxide (GO) suspension, obtained through modified Hummer's method^[13], inside a hydrophobic container such as polytetrafluoroethylene (PTFE) and heated, and GO flakes dispersed homogeneously in the GO suspension start to approach individual fibers. In the layering stage, GO flakes start depositing on individual fibers with the evaporation of volatile liquids at a temperature of 80°C. Once fully dried, fibers are then transferred into a reducing agent solution of hydrogen iodide for half an hour and washed with DI water until a pH close to 7 is obtained. This process results in cladding of rGO on each individual fiber without forming inter-fibrillar scales as shown in the photographic images of non-woven polyester fiber mat in Figure 1. The reason for the preferential deposition of rGO flakes onto the fiber rather than drying off on the substrate or forming scales is that the fiber surface, more hydrophilic than the substrate, acts as the preferential site of deposition where the rGO flakes settle and stack in a layer-by-layer fashion. The process was applied to aramid fibers (Kevlar®), polyester, cotton, nylon, wool, and glass fibers in order to show its adaptability for a variety of material systems. The former four types of samples used are in mat form, while the latter two in fiber form. The images of these fibers before and after cladding are shown in Figure 1. With 13 S/cm and 4.5 S/cm, the maximum electrical conductivity is demonstrated by the aramid and the nylon fibers, respectively, whereas the lowest electrical conductivities of 10 mS/cm and 40 mS/cm were obtained for rGO cladded wool and cotton. A table showing the electrical conductivities of a range of fibers and fiber mats before and after rGO cladding can be found in the Electronic Supplementary Information (ESI). Electrically conductive glass

and aramid fibers can potentially bridge the gap between the insulating glass and aramid and carbon fibers as the aramid and glass fibers are both tougher and less expensive than the popular carbon fibers whereas the carbon fibers are electrically highly conductive as opposed to the insulating glass and Aramid fibers.

The morphological studies of some fiber samples using scanning electronic microscope (SEM) are shown in Figure 2. Figure 2a shows polyester fibers cladded with rGO and the wrinkled structure of rGO can be clearly seen in the inset, which is a close-up image of a single polyester fiber surface. Figure 2b is a SEM image of pristine polyester fibers and the plain surface of a single polyester fiber can be seen in the inset. The cross-sectional SEM images of individual polyester and glass fibers in Figures 2c and 2d evidently demarcate the cladded surface of the fibers and the core. The inset in Figure 2c shows the wrinkled structure of rGO whereas the layered structure of rGO at the edge of a fractured glass fiber is shown in Figure 2d. SEM images of all other samples are included in the ESI.

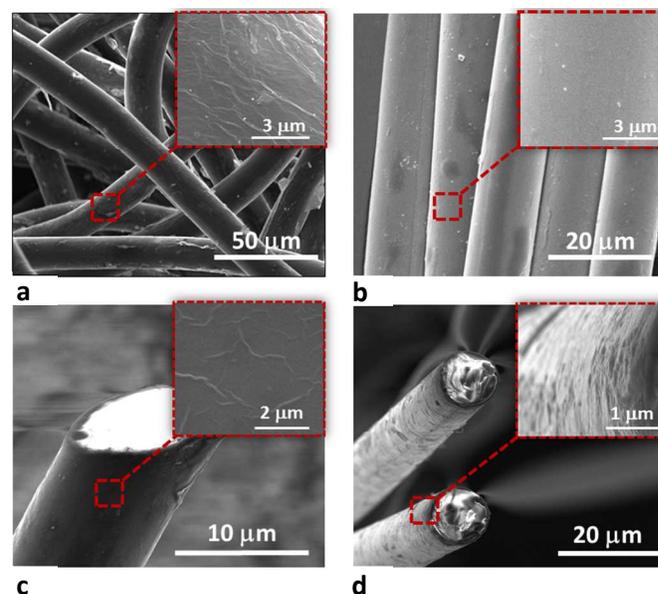


Figure 2: SEM images of rGO cladded polyester fibers and surface morphology of an individual cladded fiber (a), pristine polyester fibers and the surface morphology of an individual fiber (b), cross-sectional view of rGO cladded polyester fiber and its surface morphology (c) and cross sectional view of fractured rGO cladded glass fibers and the layered structure of rGO at the edge (d)

The rGO cladding on the aforementioned fibers and fiber mats is non-destructible by washing in detergent and sonication. Each sample was immersed for fifteen minutes in 20 weight percent (wt%) of a commercially available detergent dissolved in DI water and the results are shown as electrical conductivity vs. the wash cycle in Figure 3a. Washing was repeated for at least five times for each sample. In all the cases there is a minor drop in electrical conductivity after the first wash and subsequently there is no significant drop in any of the samples tested. However, the drop in the electrical conductivity of nylon and aramid is greater than other samples where Nylon drop from 5 to 0.5 S/cm and aramid drop from 13 to 11 S/cm. Photographs of glass fibers before and after five wash cycles are shown in the insets of Figure 3a. Similarly, all the samples were sonicated with a frequency of 40 kHz at 300W for five minutes in one sonication cycle and for at least five times and

the results are plotted as electrical conductivity vs. sonication time (Figure 3b). In the first sonication cycle all the samples show a minor drop in the electrical conductivity and then remain constant except for the aramid fibers which keep dropping to 8 S/cm until the last cycle of sonication. The drop in the first cycle is due to the loss of the loose rGO particles and some undesirable inter-fibrillar scales which once removed account for a minor drop in the electrical conductivity. Nonetheless, after the first cycle there is no significant drop in the electrical conductivity, demonstrating that the rGO cladding on the material is non-destroyable by the process. In addition to these studies, the effect of GO concentration on the electrical conductivities of Nylon and Polyester were also studied and are shown in Figure 4 in ESI.

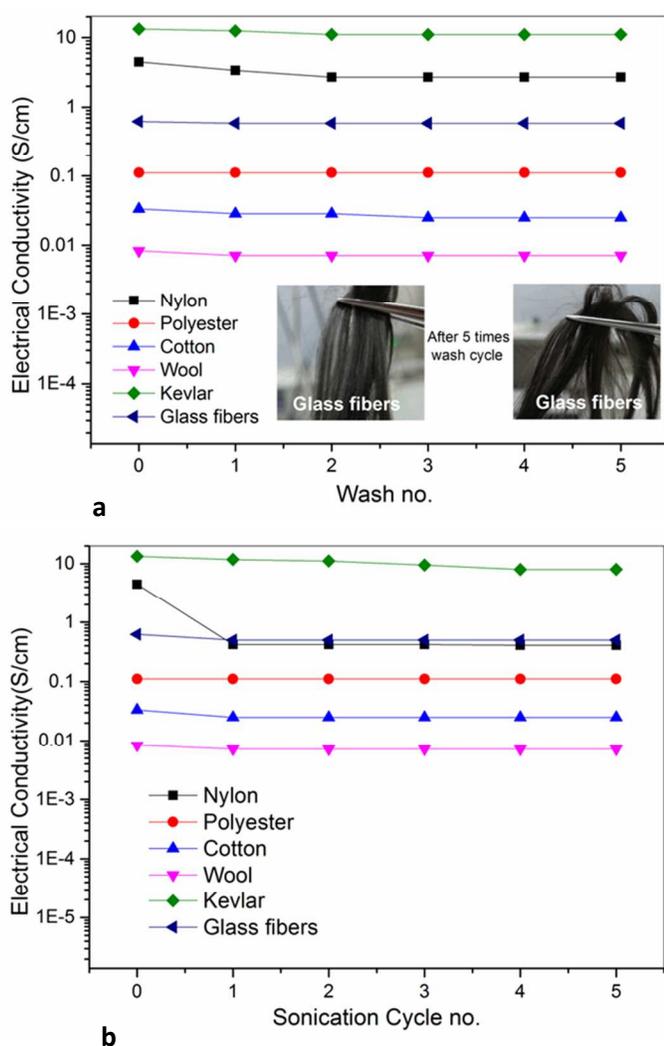


Figure 3: Electrical conductivity of fiber and fiber mat specimen with washing cycle (a) and sonication cycle (b). Connecting line only show data trend.

Conclusions

In this study a range of fibers and fiber mats were cladded with rGO via a facile cladding technique to induce electrical conductivity. Electrical conductivities of about 13 S/cm and 5 S/cm were obtained for otherwise electrically insulating aramid and nylon fiber mats, respectively. The cladding to the said fibers is shown to be uniformly assembled and non-destroyable

by washing and sonication. These electrically conductive textile and fibers can potentially play a vital role in implementing the technologies of wearable electronic devices and harvesting energy from movable resources such as human body amongst others.

Notes and references

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Electronic Supplementary Information (ESI) available: The supporting information along with this article includes the information about materials and methods, table showing the electrical conductivities of a range of fibers and fiber mats before and after cladding, SEM images of the rGO cladded fibers and an AFM image showing single layer graphene oxide flakes. DOI: 10.1039/c000000x/

[1] a) Z. Chen, W. Ren, L. Gao, B. Liu, S. Pei and H.-M. Cheng, *Nat Mater* **2011**, *10*, 424-428; b) L. Hu, M. Pasta, F. L. Mantia, L. Cui, S. Jeong, H. D. Deshazer, J. W. Choi, S. M. Han and Y. Cui, *Nano Letters* **2010**, *10*, 708-714; c) G. A. Gelves, M. H. Al-Saleh and U. Sundararaj, *Journal of Materials Chemistry* **2011**, *21*, 829-836; d) R. F. Service, *Science* **2003**, *301*, 909-911.

[2] M. Pasta, L. Hu, F. La Mantia and Y. Cui, *Electrochemistry Communications* **2012**, *19*, 81-84.

[3] a) G. Mattana, P. Cosseddu, B. Fraboni, G. G. Malliaras, J. P. Hinstroza and A. Bonfiglio, *Organic Electronics* **2011**, *12*, 2033-2039; b) I. Wistrand, R. Lingström and L. Wågberg, *European Polymer Journal* **2007**, *43*, 4075-4091.

[4] a) J. B. Lee and V. Subramanian, *Electron Devices Meeting, 2003. IEDM '03 Technical Digest. IEEE International* **2003**, pp. 8.3.1-8.3.4; b) J. Huang, H. Zhu, Y. Chen, C. Preston, K. Rohrbach, J. Cumings and L. Hu, *ACS Nano* **2013**, *7*, 2106-2113.

[5] A. G. Avila and J. P. Hinstroza, *Nat Nano* **2008**, *3*, 458-459.

[6] a) Y. Liu, J. Tang, X. Chen and J. H. Xin, *Carbon* **2005**, *43*, 3178-3180; b) Y. Liu, J. Tang, R. Wang, H. Lu, L. Li, Y. Kong, K. Qi and J. H. Xin, *Journal of Materials Chemistry* **2007**, *17*, 1071-1078.

[7] a) Y. K. Ramadass and A. P. Chandrakasan, *Solid-State Circuits, IEEE Journal of* **2011**, *46*, 333-341; b) O. Bubnova, Z. U. Khan, A. Maiti, S. Braun, M. Fahlman, M. Berggren and X. Crispin, *Nat Mater* **2011**, *10*, 429-433.

[8] J. Bae, M. K. Song, Y. J. Park, J. M. Kim, M. Liu and Z. L. Wang, *Angewandte Chemie International Edition* **2011**, *50*, 1683-1687.

[9] V. Leonov, *Sensors Journal, IEEE* **2013**, *13*, 2284-2291.

[10] a) R.-H. Kim, M.-H. Bae, D. G. Kim, H. Cheng, B. H. Kim, D.-H. Kim, M. Li, J. Wu, F. Du, H.-S. Kim, S. Kim, D. Estrada, S. W. Hong, Y. Huang, E. Pop and J. A. Rogers, *Nano Letters* **2011**, *11*, 3881-3886; b) T. Aida, I. Murayama, K. Yamada and M. Morita, *Journal of Power Sources* **2007**, *166*, 462-470.

[11] a) A. A. Balandin, S. Ghosh, W. Bao, I. Calizo, D. Teweldebrhan, F. Miao and C. N. Lau, *Nano Letters* **2008**, *8*, 902-907; b) C. Soldano, A. Mahmood and E. Dujardin, *Carbon* **2010**, *48*, 2127-2150.

[12] S. Stankovich, D. A. Dikin, R. D. Piner, K. A. Kohlhaas, A. Kleinhammes, Y. Jia, Y. Wu, S. T. Nguyen and R. S. Ruoff, *Carbon* **2007**, *45*, 1558-1565.

[13] X. Li, G. Zhang, X. Bai, X. Sun, X. Wang, E. Wang and H. Dai, *Nat Nano* **2008**, *3*, 538-542.

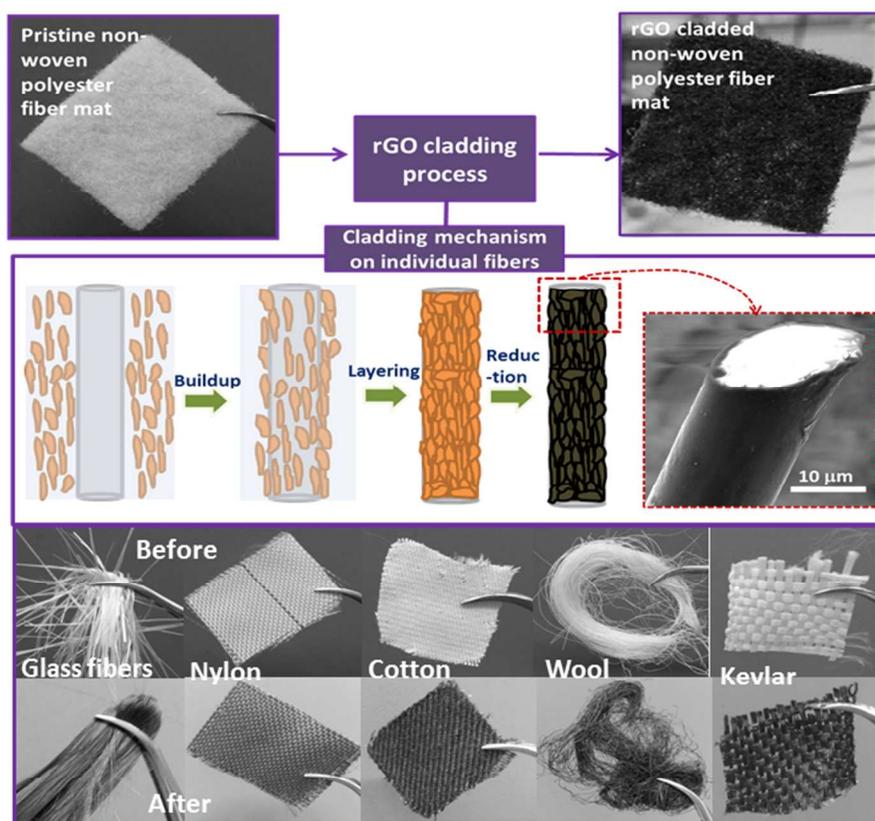


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