

Thin Films and Coatings

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New Low Voltage FE-SEM & AFM Techniques Offer Unprecedented Glimpse into Graphene

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Low voltage (<1kV) field emission scanning electron microscopy (LV-FESEM) provides a high quality easy to use imaging technique for energy-sensitive materials whilst still maintaining high imaging resolution. For non-conducting samples, the application of a thin conducting surface coating can sometimes be eliminated when a state of charge equilibrium is achieved on the specimen's surface and this approach has been successfully applied to graphene films deposited onto insulating substrates. It should be noted that applying a conductive coating can compromise with the imaging of graphene and is therefore not recommended.

The presentation will show low voltage imaging of graphene on a range of substrates. Secondary electron, backscattered electron and topographic imaging modes were used to study the surface morphologies of graphene samples at a broad range of magnifications. Reducing the incident beam voltage to 500 volts is shown as an effective method in controlling the electron charge caused by insulating substrates beneath single and multiple graphene layers. It will be demonstrated, with examples, that LV-FESEM imaging can successfully be employed in graphene-related research and can further be applied to quality checks, determining the number of graphene layers deposited on a substrate and visualizing impurity particles sometimes present in engineered graphene manufacture. [1-4].

High-resolution visualization of the surface morphology is a key feature of the Atomic Force Microscopy (AFM) technique and the capability of routinely resolving details at a single molecular level has been well demonstrated previously. Furthermore advanced AFM methods can be used to characterize the electrical properties of surfaces. In this presentation we describe Kelvin Force Microscopy (KFM) which is a single pass surface potential measurement. KFM offers a powerful experimental means to investigate the local electrical properties of both single layer graphene and few layer graphene films. The effect of the film thickness on the surface potential is detected and quantitative measurements are obtained. [5-6].

1. K.S. Novoselov et al, Science 306, (2004) p. 666.
2. F. Schwierz, Nat. Nano. 5, (2010) p. 487.
3. Y.-M. Lin et al, Science 327, (2010) p. 662.
4. J. Hass et al, J. Phys: Condens Matter 20, (2008) p. 323202.
5. S. Zhou, G.-H et al, Nat. Mat. 6, (2007) p. 770.
6. A. Bostwic et al, Nat. Phys. 3, (2007) p. 36.

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Nanocrystal orientation and superlattice array in Pbs-oleic acid thin films: The role of crystal habit and organic fibrils

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With regard to the materials science aspects of nanocolloid structural chemistry, this study is of great interest in the attempt to answer two of the major topics being hotly debated. The first concerns the principles of the ordering of self-assembled films and the driving forces for this kind of self-assembly [1,2]. The second topic concerns itself with the optimisation of physical properties of such systems containing confined and isolated but nevertheless interconnected structures [3].

For the self-assembly processes of ligand capped nanoparticles a complex set of events should be taken into account, such as the attraction between the surface charges of the inorganic nanoparticles and a significant contribution from van der Waals forces between the organic surface chains which are further assisted by capillary forces evolving during the drying processes and steric effects caused by the shape of the individual nanoparticles. As soon as a structure has been established by the self assembly process, further ordering may be assumed to be prevented by fibrillation of the organic surfactant. By careful analysis of high-resolution bright field images it was found that the nanoparticles are interconnected by organic fibrils of oleic acid which are partially mineralised by PbS. These inorganic bridges on an atomic level together with organic fibrils are responsible for keeping the orientation of the nanoparticles fixed thus preventing the system from adopting a disordered or arbitrary arrangement. Recently, we were able to determine the 3D structure of a PbS - oleic acid superassembly showing an intermediate character between a high and low orientational ordering of nanoparticles, respectively [4]. In this system, the truncated octahedrally shaped PbS nanocrystals are stabilized by organic molecules and assembled into an fcc superlattice with long-range "translational" order, while still maintaining their preferable crystallographic orientation limited to shorter ranges. This situation is reminiscent of a special type of colloidal crystal classified as "mesocrystals" which represent superstructures of nanoparticles with certain mutual orientation relations

1. S. A. Harfenist, Z. L. Wang, M. M. Alvarez, I. Vezmar, R. L. Whetten. *J. Phys. Chem* (1996) 100, 13904-13910.
2. Z. L. Wang, S. A. Harfenist, I. Vezmar, R. L. Whetten, J. Bentley, N. D. Evans, K. B. Alexander. *Adv. Mater.* (1998) 10, 13-30.
3. D. V. Talapin, J.-S. Lee, M. V. Kovalenko, E. V. Shevchenko. *Chem. Rev.* (2010) 110, 389–458.
4. P. Simon, E. Rosseeva, I. A. Baburin, L. Liebscher, S.G. Hickey, R. Cardoso-Gil, A. Eychmüller, R. Kniep, W. Carrillo-Cabrera. *Angew. Chem. Int. Ed.* (2012) 51, 10776-10781.
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