

1: Non-destructive imaging of buried electronic interfaces using a decelerated scanning electron beam

Radiation technologies (ion implantation, ion beam mixing, plating, etc.) are powerful techniques in the modification of surface and near-surface properties of solids. Although they hold great.

Supplementary data Comprehensive understanding of biological objects—their chemical, physiochemical and biological characteristics—can be effectively achieved through electron microscopy EM analysis [1 – 4], preferably without any fixation or auxiliary surface treatment. Recently, EM analysis has extended its use to construct three-dimensional structure of the biological specimen with combination of serial block-face sectioning or focused ion beam [7 , 8]. In addition, the unique interaction between electron beams and specimen enables various physical and chemical analyses such as energy dispersive spectroscopy EDS , electron probe micro analysis, and electron energy loss spectroscopy [9 , 10]. Nevertheless, charge accumulation by electron beams and shrinkage of samples by dehydration in vacuum have always hindered EM-mediated biological studies as it distorts the morphological and chemical characteristics of the specimens [11 – 16]. For these reasons, various coating and sample preparation methods for EM analysis have been developed to enhance the image contrast from non-conducting biological specimen [1 , 5 , 17 – 28]. In particular, a metal- and carbon-coating methods have been widely employed to dissipate the accumulated charges on non-conducting surface [23 – 27]. However, the relatively thick coating layer hampers from studying the fine structures of the specimen at nanometer scale because of the large size of metal or carbon grains. In addition, x-ray fluorescence signals required for EDS analysis are screened by metal layers [29]. Furthermore, it is usually difficult to use the metal-coated samples for further analyses such as TEM that requires electron-transparency. Recent progresses in large scale synthesis of high quality graphene films using chemical vapor deposition CVD methods have widened its potential in practical device applications as well as unique interests in basic scientific researches [33 – 36]. The feasibility of the large scale fabrication of continuous graphene films as well as easy transfer onto diverse biological objects opens up a unique opportunity to create new hetero-interfaces or interfaces with non-conducting biological samples. As demonstrated in a recent work [37], the in situ high-resolution EM imaging of nanocrystal growth has been achieved by using graphene liquid cells to encapsulate nanoscale materials as well as their environment i. In this regard, graphene mediated coating on biological samples can provide high-resolution EM imaging and chemical analysis due to the excellent electron and heat flow thorough the graphene and electron-transparency [44]. Here, with taking all these advantages of graphene films, we have employed continuous graphene films as coating for biological samples and exploited them for non-destructive high-resolution EM imaging and chemical analysis. As schematically displayed in figure 1 , the unique feasibility and availability of continuous graphene films at large scale enables the conformal coating of biological objects including leaves, ants, spiders, neuron cells, Escherichia coli E. Atomically-thin and electrically-conducting graphene membranes were prepared on non-conducting biological surface by isolating graphene films from copper Cu foils after CVD growth, followed by conformal coating onto biological samples as illustrated in figure 1 c. Compared to other conventional sample preparation methods including fixation and metal sputter coating figures 1 a and b , the present method based on graphene coating is relatively simple, bio-friendly and non-destructive, which is particularly advantageous for preserving the chemical information of samples for further experiments. Schematic illustration of various biological objects in different scales and coating methods for EM analysis. Soft biological samples such as cells and bacteria require complicated coating processes including aldehyde fixation, osmium tetroxide fixation, dehydration, critical point drying, staining, metal coating, etc. Hard-surfaced biological samples such as insects and plants are usually coated with Au, Pt by vacuum sputtering. The metal coating is simple, but it disables high-resolution imaging and analysis. The ambient drying process allows the conformal coating of graphene on sample surface. Standard image High-resolution image Export PowerPoint slide Monolayer graphene film was synthesized on high-purity Cu foil using CVD method please see supplementary materials. Continuous graphene films coated with a poly methyl methacrylate PMMA layer can be isolated from Cu foils and transferred to a target surface after wet chemical

etching [35]. The number of graphene layers was controlled by repeating this transfer process. We found that triple-layered 3-layer graphene films provide optimum electron transparency and mechanical stability for SEM analysis figures S1–S3. The biological specimens were cleaned and positioned onto a metallic sample stage for SEM imaging. The 3-layer graphene sheets were then transferred on top of the biological specimen by scooping from bottom side, followed by drying in a desiccator. The 3-layer graphene mostly covered these millimeter to nanometer sized samples, and only shows minor fractures around needle-like structures figure 2 c. In contrast, the use of graphene oxide GO and reduced GO resulted in incomplete coating due to their poor mechanical strength and difference in hydrophobicity figure S4. The high-magnification FE-SEM images of a graphene-coated ant clearly show not only unique micro-patterns but also nano-pores as small as 40 nm figure 2 b that are invisible in platinum Pt -coated or carbon-coated samples figures 2 i and S5. Such fine and clear observation of the surface structures implies that the adhesion between graphene and the sample mostly by van der Waals interaction is strong enough to maintain its morphology [38] and stable up to acceleration voltage of 20 kV figure S6. We also performed SEM imaging on a 1. High-magnification SEM images of the water flea area P1 in figure 2 d clearly display the unique features of a water flea on its dorsal carapace figure 2 e. Interestingly, the graphene film mostly covers the needle-like surface on its antenna without much tearing figure 2 f. SEM images of various biological samples covered with graphene films. The graphene film exhibits conformal contact with the non-flat surfaces of biological samples. Acceleration voltages for A to F, 2 kV. The graphene coating enables the stable SEM imaging of sub nm features on the surface, while the Pt-coated sample shows distorted morphology covered with Pt nanoparticles. Standard image High-resolution image Export PowerPoint slide The advantages of graphene coating compared with a conventional metal coating method were demonstrated under identical conditions figures 2 g – i. Unlike the above mentioned hard-surfaced insects, soft biological objects such as tissues, cells and bacteria need an additional treatment for EM analysis, including aldehyde fixation, osmium tetroxide staining, and critical point drying. In this regard, the simple graphene-coating method can be advantageous because biological samples close to their native structures can be imaged and preserved for further analyses. If combined with conventional fixation methods, it would be more effective for the high-resolution EM imaging of biological samples figure S7. We also demonstrate that common bacteria, E. Recently, an environmental SEM ESEM has been utilized to observe the native structures of biological samples without conductive coating, but its resolution is still limited due to the charging problem associated with low conductivity of biological surfaces figures S7 and S8 [39 – 41]. We also observed that untreated E. On the other hand, the graphene coating not only provides higher resolution than ESEM but also stabilizes the liquid-containing samples that can be easily damaged by intense electron beams. We also performed EM imaging of ferritin, an intracellular protein that stores and releases iron to control the iron concentration in living organisms [42]. The ferritin particles in water are encapsulated between two monolayer graphene films. The individual ferritin particles are clearly observed in SEM, in which the iron cores look brighter figure 2 m. In a low-magnification TEM image, spherical protein shell as well as hydrous ferric oxide cores were identified from their different contrast, and at high-magnification, the lattice fringe of the iron core was clearly resolved with atomic resolution in an aqueous medium figure 2 o. Likewise, we also demonstrate that the hydrated structure of plasmid deoxyribonucleic acids of E. We compared the performances of graphene-coating and Pt-coating methods in chemical analysis by EDS. All the experimental conditions and parameters including spot sizes and signal collection time were identical. The results showed that EDS signals from graphene-coated samples figure 3 a are 2–3 times stronger than Pt-coated samples figure 3 c , which facilitates the qualitative and quantitative chemical analyses on nitrogen-containing chitin from ants and oxygen-rich cellulose from leaves. The non-destructive analysis enabled by the graphene coating is particularly efficient for element-specific EDS mapping. The water flea sample was fed with 25 nm cerium oxide nanoparticles CeO₂ NPs to stain its digestive pathway please see supplementary materials for experimental details. The other EDS analyses also indicate that the graphene-coated method is superior to Pt-coating in terms of signal intensity figure S We attribute the signal reduction in the Pt-coated samples to the absorption and scattering of incident electrons and x-ray fluorescence radiation by thick Pt layers, which will be further discussed in figure 4. Acceleration voltages, 10 keV.

Acceleration voltages, 20 keV. The corresponding EDS spectra are shown in supplementary figure S

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Elemental depth profiles with a depth resolution of 5 - 50 nanometers and a maximum depth of 2 - 20 microns. Surface impurities and impurity distribution in depth sensitivity up to sub-ppm range. Elemental areal density and thus thickness or density of thin films if the film density or thickness is known. Diffusion depth profiles between interfaces up to a few microns below the surface. Channeling-RBS is used to determine lattice location of impurities and defect distribution depth profile in single crystalline samples Forward recoil spectrometry FReS: Nondestructively and simultaneously determines hydrogen isotopes H and D and their depth profiles in polymers and other solids with a sensitivity of 0. Nuclear reaction analysis NRA: Unlike ion scattering techniques, NRA is an isotopically sensitive technique with an excellent mass resolution and has no mass-depth ambiguity of RBS and FReS in data interpretation. Channeling-NRA can be used to determine lattice location of impurities and defect distribution depth profile in single crystalline samples. Nondestructive and multielemental analysis of trace elements with an excellent detection limit of up to 20 ppb. Used together with RBS for accurate mass identification of medium to heavy elements with similar masses. Channeling-PIXE can be used to determine lattice location of impurities in single crystalline samples Ion channeling analysis Assess crystallinity of MBE-grown thin films such as type of defect structures, impurity location, type of atomic site, lattice strain and alignment in epitaxial growth Enhance surface sensitivity of light elements on heavier single crystal substrate Channeling-RBS, Channeling-NRA, and Channeling-PIXE are available for different applications Specifications: Accelerator terminal voltage tunable from 80 kV to 1. Beam spot size from 0. Beam current on target up to a few tens to hundreds nA depending on ion species and energies. Ortec Ultra ion detectors: Canberra 2" x 2" NaI Tl gamma detector: Sample has to be a vacuum-compatible solid with reasonably smooth surface. The above compares data taken at random sample orientation unchanneled and under axial channeling conditions. The colored arrows denote the energies of backscattered He nuclei from different elements at two depth locations. Protocol for analytical services in IBA lab Industrial clients: Often includes computer simulation of anticipated spectra, given rough description of usually layered samples. Agree on scope of work: Estimate of price including post-processing of data to the degree suggested in the previous bullet. Mail or drop off samples lab combination can be provided in IN tray just inside of lab, at left; work usually proceeds within work days. Data is immediately emailed to client following acquisition, if no post processing is required. The latter typically would be conducted within work days depending on analytical complexity, number of spectra, and other staff commitments. Optional user-conducted data analysis may require training:

3: Semiconductor Failure Analysis (FA) Techniques

Non-Destructive Surface Analysis of Materials by MeV Ion Beams, Microscopy and Computer Simulation - Volume 22 Issue S4 - J. Pacheco de Carvalho, C. F. R. Pacheco, A. D. Reis Skip to main content We use cookies to distinguish you from other users and to provide you with a better experience on our websites.

History[edit] In British physicist J. Thomson observed a release of positive ions and neutral atoms from a solid surface induced by ion bombardment. Castaing for the PhD thesis of G. In the s, K. Magee developed SIMS instruments equipped with quadrupole mass analyzers. S and used in materials science and surface science. High energy usually several keV ions are supplied by an ion gun 1 or 2 and focused on to the target sample 3 , which ionizes and sputters some atoms off the surface 4. These secondary ions are then collected by ion lenses 5 and filtered according to atomic mass 6 , then projected onto an electron multiplier 7, top , Faraday cup 7, bottom , or CCD screen 8. A secondary ion mass spectrometer consists of 1 a primary ion gun generating the primary ion beam , 2 a primary ion column, accelerating and focusing the beam onto the sample and in some devices an opportunity to separate the primary ion species by Wien filter or to pulse the beam , 3 high vacuum sample chamber holding the sample and the secondary ion extraction lens, 4 a mass analyser separating the ions according to their mass-to-charge ratio, and 5 a detector. This is needed to ensure that secondary ions do not collide with background gases on their way to the detector i. Primary ion source[edit] Three types of ion guns are employed. This type of ion gun is easy to operate and generates roughly focused but high current ion beams. Depending on the gun design, fine focus or high current can be obtained. A third source type, the liquid metal ion gun LMIG , operates with metals or metallic alloys, which are liquid at room temperature or slightly above. The liquid metal covers a tungsten tip and emits ions under influence of an intense electric field. While a gallium source is able to operate with elemental gallium, recently developed sources for gold , indium and bismuth use alloys which lower their melting points. It is therefore commonly used in static SIMS devices. The choice of the ion species and ion gun respectively depends on the required current pulsed or continuous , the required beam dimensions of the primary ion beam and on the sample which is to be analyzed. Oxygen primary ions are often used to investigate electropositive elements due to an increase of the generation probability of positive secondary ions, while caesium primary ions often are used when electronegative elements are being investigated. A sector field mass spectrometer uses a combination of an electrostatic analyzer and a magnetic analyzer to separate the secondary ions by their mass-to-charge ratio. A quadrupole mass analyzer separates the masses by resonant electric fields, which allow only the selected masses to pass through. The time of flight mass analyzer separates the ions in a field-free drift path according to their velocity. Since all ions possess the same kinetic energy the velocity and therefore time of flight varies according to mass. It requires pulsed secondary ion generation using either a pulsed primary ion gun or a pulsed secondary ion extraction. It is the only analyzer type able to detect all generated secondary ions simultaneously, and is the standard analyzer for static SIMS instruments. Detector[edit] A Faraday cup measures the ion current hitting a metal cup, and is sometimes used for high current secondary ion signals. With an electron multiplier an impact of a single ion starts off an electron cascade, resulting in a pulse of electrons which is recorded directly. A microchannel plate detector is similar to an electron multiplier, with lower amplification factor but with the advantage of laterally-resolved detection. Usually it is combined with a fluorescent screen, and signals are recorded either with a CCD-camera or with a fluorescence detector. Detection limits and sample degradation[edit] Detection limits for most trace elements are between and atoms per cubic centimetre , [12] depending on the type of instrumentation used, the primary ion beam used and the analytical area, and other factors. Samples as small as individual pollen grains and microfossils can yield results by this technique. While only charged secondary ions emitted from the material surface through the sputtering process are used to analyze the chemical composition of the material, these represent a small fraction of the particles emitted from the sample. Static SIMS is the process involved in surface atomic monolayer analysis, or surface molecular analysis, usually with a pulsed ion beam and a time of flight mass spectrometer, while dynamic SIMS is the process involved in bulk analysis, closely related to the sputtering

process, using a DC primary ion beam and a magnetic sector or quadrupole mass spectrometer. Applications[edit] The COSIMA instrument onboard Rosetta will be the first instrument to determine the composition of cometary dust in situ with secondary ion mass spectrometry in

4: The Center for Advanced Life Cycle Engineering

Ion beam analysis (IBA): using MeV ion beams for compositional and structural determination of materials, combines the advantages of non-destructive and standardless analysis of the surface and near surface regions (microns) of solids.

5: Ion Beam Analysis Techniques - RBS Lab

This comprehensive tutorial presentation not only reviews the fundamental features of rapid nuclear analysis methods (Rutherford backscattering and channelling, in conjunction with changes of ion energy, ion-induced X-ray emission, nuclear microanalysis); it also presents original experimental results.

6: Non-destructive Ion Beam Analysis of Art Objects - Helmholtz-Zentrum Dresden-Rossendorf, HZDR

Book Review: Non-destructive ion beam analysis of surfaces. by F.F. Komarov, M.A. Kumakhov and I.S. Tashlykov (Gordon and Breach Science Publishers, New York,) pp. xiv + ,(book club) CIP.

7: Atmosphere influence on in-situ ion beam analysis of thin film growth – Northwestern Scholars

The application of ion-beam surface-analysis techniques to plasma-edge studies in fusion devices is reviewed. Methods of ion beam analysis are described for the quantitative determination of the species, concentration, and depth distribution of atoms collected on surface probes or other exposed surfaces in the plasma edge.

8: Secondary ion mass spectrometry - Wikipedia

Non-destructive ion beam analysis of surfaces Book Komarov, F.F. ; Kumakhov, M.A. ; Tashlykov, I.S. Radiation technologies (ion implantation, ion beam mixing, plating, etc.) are powerful techniques in the modification of surface and near-surface properties of solids.

9: Department of Chemistry : Ion Beam Analysis - Durham University

In particular, non-destructive ion-beam chemical analyses (PIXE and PIGE) have been performed on 11 ceramic artefacts from the Cleveland Museum of Art (CMA) and a like number from several French.

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