

Wednesday Afternoon Poster Sessions, December 5, 2018

Nanomaterials

Room Naupaka Salon 1-3 - Session NM-WeP

Nanomaterials Poster Session II

Moderator: Paul Stoddart, Swinburne University of Technology

NM-WeP1 Fabrication of Morpho Butterfly Structure using Standing Wave Effect, *Tomoki Nishino, H. Tanigawa*, Ritsumeikan University, Japan; *A. Sekiguchi*, Litho Tech Japan Corporation, Japan

In recent years, biomimetic technologies have been extensively studied. Morpho butterflies are representative examples of structural colors, and many scientists have done research. On the wing, small thin plates called scales with a size of 70 x 150 μm and a thickness of a few microns are spread like roof tiles. When one scaled powder is enlarged, it has a streaky structure on the scale, this streak is called ridge. It is a characteristic of morpho butter that each of the ridges (lid interval: 1 μm) appears blue.

In this paper, the morpho structure can be fabricated by using lithography technology. The standing wave effect is exposure unevenness generated in the resist film when the photoresist on the Si substrate is exposed with monochromatic exposure light. Two reflected lights of the resist surface and the Si substrate cause interference waves. It strengthens and weakens according to the film thickness. No interference wave is generated by the reflected light of the Si substrate, and constant reflection intensity of light is always obtained.

Patterning was performed on the resist, cross-sectional development was performed, and the standing wave effect in the resist film was confirmed.

We propose that the morpho structure can be fabricated by using lithography technology.

NM-WeP2 Photovoltaic Performance of Organic Polymer Solar Cells using Silicon Nanoparticles with Various Phosphorus Contents, *Naaki Ikeda, K. Sato*, Tokyo Denki University, Japan

Silicon nanoparticles (SiNPs) are used for solar cell applications because they not only enhance light absorption efficiency of the photoelectric conversion layer but also expand the absorption bands of sunlight. We have fabricated the phosphorus (P)-doped SiNPs using an electroless chemical etching, which is inexpensive and simple techniques. When the P powders was used as n-type dopant, it was difficult to uniformly incorporate the P atoms in the SiNP core. To overcome this challenge, we performed the P doping into SiNPs using P dispersed solvent. In this presentation, we report the photovoltaic performance of the organic polymer solar cells using SiNPs with various P contents. The P-doped SiNPs were fabricated by etching the Si powders with a diameter of 100 nm in a hydrofluoric acid/nitric acid mixture solution using high-speed mixer and by thermally diffusing the P atoms at high temperature. The P contents in the SiNPs were controlled by varying the annealing temperature. The solar cells were prepared by individually coating the P-doped SiNPs and poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS) on the micro-pyramidal structured Si substrate. The current density-voltage characteristics of undoped SiNPs/PEDOT:PSS solar cells and the P-doped SiNPs/PEDOT:PSS solar cells were shown in Figure 1. The solar cell using undoped SiNPs showed a power conversion efficiency (PCE) of 4.81% with a short circuit current density (J_{sc}) of 27.7 mA/cm², an open circuit voltage (V_{oc}) of 0.361 V and a fill factor (FF) of 0.482. On the other hand, these cell parameters dramatically increased by doping the P atoms into SiNPs; that is, the values of J_{sc} , V_{oc} , FF and PCE were 29.7 mA/cm², 0.349 V, 0.560 and 5.79%. These results are attributable to the uniform incorporation of P atoms into SiNPs by the use of the P dispersed solvent and the improvement of transport efficiency of photogenerated charge carriers between the SiNPs.

NM-WeP3 Effect of Phosphorus-doping on Photovoltaic Performance of Si Nanoparticles/Polymer Hybrid Solar Cells, *Masataka Takase, K. Sato*, Tokyo Denki University, Japan

Semiconductor nanoparticles (NPs) are increasingly gaining attention as suitable materials to enhance photovoltaic performance for solar cell applications because the absorption regions of solar light can control by tuning the band gap due to quantum confinement effect. We have investigated the photovoltaic performance of solar cells using phosphorus (P) doped n-type silicon NPs (SiNPs) so as to improve the transport efficiency of photogenerated charge carriers, and we have compared to that of the undoped SiNPs. In this presentation, we report the effect of P-doping on the photovoltaic performance of solar cells using n-type silicon NPs (SiNPs). The n-type SiNPs were prepared by doping P dopant in commercially available NPs of 100 nm in size using thermal diffusion

process at high temperature. The doping states of P atoms in the obtained n-type SiNPs were confirmed by X-ray photoelectron spectroscopy (XPS), indicating that the P atoms exist as the P-Si bonds at the substitutional sites of Si atoms in the crystalline core of n-type SiNPs. We prepared the solar cells in which the undoped SiNPs layer or n-type SiNPs layer applied on the micro-pyramid-shaped Si substrate was covered with poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS), which is p-type conducting polymers. The solar cells using the undoped SiNPs showed the low power conversion efficiency (PCE) of 1.44% with open circuit voltage (V_{oc}) of 0.318 V, short circuit current density (J_{sc}) of 10.1 mA/cm² and fill factor (FF) of 0.451. When the active layer in the solar cells replaced the undoped SiNPs by the n-type SiNPs, the values of PCE, J_{sc} and FF dramatically increased to 4.79%, 22.3 mA/cm² and 0.520, respectively. These improvements are because the reduction in the value of the series resistance (R_s) (0.23 $\Omega\cdot\text{cm}^2$ for the solar cells using the n-type SiNPs) due to the increase in the number of photogenerated charge carriers by P doping. This result demonstrates that the P-doped n-type SiNPs is beneficial for obtaining the higher PCE.

NM-WeP4 Effect of Amino Modification on Photovoltaic Performance of Silicon/Polymer Solar Cells with Porous Desert Structures, *Kento Saito, K. Sato*, Tokyo Denki University, Japan

Various micro-texture structures have been reviewed in silicon (Si)/polymer solar cells. Our research group have suggested a facile approach to forming micro-desert structure (MDS) on the Si substrate surface by enclosing Si substrate, phosphorus (P) powders and hydrofluoric acid (HF)/nitric acid (HN) mixed solution in a sealed container. However, the MDS is confronting serious problem for antireflection effect, which affects the power conversion efficiency (PCE) of the solar cells, showing the reflectance of 29.8 % in the incident wavelength range of 500-1000 nm. To drop the reflection of the MDS, we fabricated a porous desert structure (PDS) with ordered cylindrical mesopore in the MDS by treating in a mixed solution containing silver perchlorate (AgCl)/sodium hydroxide (NaOH). Additionally, the inside wall of the mesopore of the PDS was modified with amino group to infiltrate the polymer into the mesopore. In this presentation, we report the effect of the amino modification on the photovoltaic performance of the Si/polymer (poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS)) solar cells with PDS. The PDS with the pore diameter of approx. 20 nm and the depth of approx. 500 nm was confirmed by scanning electron microscopy (SEM). Such PDS showed the low reflectance of 1.93 % or less in the incident wavelength range of 500-1000 nm, indicating an excellent antireflection effect. When the PDS was modified with amino group, the photovoltaic performance was dramatically improved. The Si/ PEDOT:PSS solar cells with the amino-modified PDS attained the PCE of 4.53% with a short-circuit current density (J_{sc}) of 27.9 mA/cm², an open-circuit voltage (V_{oc}) of 0.362 V and a fill factor (FF) of 0.450 compared to the solar cells having the unmodified PDS with the values of J_{sc} of 22.9 mA/cm², V_{oc} of 0.261 V, FF of 0.375 and PCE of 2.24 %. This is due to the fact that affinity between the Si with the PDS and the water-soluble PEDOT:PSS enhanced by the modification of hydrophilic amino group.

NM-WeP6 Indoor Light Photocatalytic Performance of Graphene Quantum Dot-TiO₂-PAN Composite based on Electrospinning Matrix, *Hyonkwang Choi, W. Yang*, KwangWoon University, Korea, Republic of Korea

TiO₂ is one of the most promising catalysts owing to its relatively low cost, superior photocatalytic performance, long-term stability, low cost, and low toxicity under UV illumination.

However, the inherent drawbacks of the TiO₂, e.g. the poor response to visible light due to its large bandgap of 3.2 eV, and the fast recombination of photogenerated electron-hole pairs, have significantly impeded the photocatalytic application. To address these matters, the absorption of TiO₂ has been extended into the visible range using chemical doping. Nitrogen doping, especially, can alter the band structure or suppress the recombination efficiency of the photogenerated electron-hole pairs, resulting in an enhanced photocatalytic capacity of TiO₂ in the visible light region.

We present a facile method to the fabrication of nitrogen-doped GQDs/TiO₂ via polyacrylonitrill (PAN)-based carbon fibers (PAN-CFs). Electrospun PAN-CFs have here been selected to produce an electrochemically-active carbon-network matrix containing in-frame incorporated nitrogen, because of its high carbonization yield and high controllable residual nitrogen content. Also, an improvement in visible light absorption is enabled at the GQDs/TiO₂ heterostructure due to bandgap narrowing of TiO₂ by Ti-O-C bond formed between GQDs and TiO₂. PAN

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plays a dual role, nitrogen doping during its cyclization as well as acting an electrical conducting substrate of a support that enhances the performance of other decorated active catalysts. Thus, nitrogen-doping process of GQDs/TiO₂ heterostructure and its corresponding structural and optical properties were investigated through a series of experiments (SEM, TEM, FTIR, XPS, XRD, UV-Vis, PL). We also demonstrate that GQDs/TiO₂/PAN electrodes exhibit enhanced photochemical and electrochemical activities advantageous by indoor light methylene blue test and solar-driven hydrogen evolution reaction.

NM-WeP7 Multispectral Optical Imaging Retrofitted to XPS and SIMS Instruments, Peter Cumpson, I. Fletcher, N. Sano, Newcastle University, UK
All X-ray Photoelectron Spectroscopy (XPS) and Time-of-Flight Secondary Ion Mass Spectrometry (ToFSIMS) instruments have optical cameras to image the specimen under analysis, and often to image the sample holder as it enters the system too. These cameras help the user find the appropriate points for analysis of specimens. However they seldom give as good images as stand-alone bench optical microscopes, due to the limited geometry, source/analyser solid angle and Ultra-High-Vacuum (UHV) design compromises. This often means that the images displayed to the user necessarily have low contrast, low resolution and poor depth-of-field.

To help identify the different regions of the samples present we have found it useful to perform multispectral imaging by illuminating the sample with narrow-wavelength-range light emitting diodes (LEDs). By taking an image under the illumination of these LEDs in turn, each at a successively longer wavelength, one can build-up a set of registered images that contain more information than a simple Red-Green-Blue image under white-light illumination. We show that this type of multispectral imaging is easy and inexpensive to fit to common XPS and ToFSIMS instruments, using LEDs that are widely available. In our system we typically use 14 LEDs including one emitting in the ultraviolet (so as to allow fluorescent imaging) and three in the near infra-red. The design considerations of this system are discussed in detail, including the design of the drive and control electronics, and three practical examples are presented where this multispectral imaging was extremely useful in XPS and SIMS.

NM-WeP8 Synthesis of Small Cubic Metal Nanoparticles of Fe⁰, Co⁰ and Ni⁰ by using Calcium Hydrate as Reducing Agent, Maria Volokhova, A. Boldin, L. Seinberg, National Institute of Chemical Physics and Biophysics, Estonia

Metal oxide nanoparticles (NPs) are widely used in different biomedical fields, such as tissue engineering [1], MRI contrast enhancement [2] and detection of proteins [3]. In the current study we are focusing on synthesizing metal nanoparticles (size ~20 nm). In comparison with their oxide counterparts, metal NPs with improved magnetic properties could be used as a MRI contrast agent. Our metal NPs were synthesized using metal oxide NPs, which have been synthesized beforehand by using simple one-pot pyrolysis [4]. Metal hydrate salt and polyvinylpyrrolidone (PVP) in DMF were used as starting materials. The reaction is carried out in a teflon tube that is heated in a furnace (< 200°C). The metal oxide NPs were covered with SiO₂ and reduced using CaH₂ in a vacuum sealed tube [5]. Therefore, NPs with a cubic metal core and a SiO₂ shell coating were obtained. The crystal structure and morphology of the NPs were analyzed using X-ray diffraction (XRD) and transmission electron microscope (TEM). Using the physical properties measurement system (PPMS), we have studied the magnetic properties of the NPs at the room temperature. As far as biomedical applications are concerned, we improved the solubility of the NPs in water. For this purpose zwitterionic dopamine sulfonate (ZDS) [6] was synthesized and used as a coating material for the NPs. Preliminary toxicology experiments of the NPs confirmed their suitability for biomedical application.

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NM-WeP10 Control of Fluorescence Color and Magnetic Intensity of Magnetofluorescent Microparticles, Takafumi Yasuzawa, K. Sato, Tokyo Denki University, Japan

In recent years, magnetofluorescent materials have attracted attention as one of a powerful tool to provide simultaneously diagnosis and treatment of cancer cells such as image diagnosis using fluorescence property and thermotherapy using magnetism property. We have developed the magnetofluorescent microparticles in which fluorescent ammonium silicon fluorides ((NH₄)₂SiF₆) were formed on the surface of the ferromagnetic magnetite (Fe₃O₄) layer, which applied on the Si substrates. This microparticles have some serious challenges; for instance, emission of only single light (red-orange color) and poor magnetic response due to the formation of the diamagnetic ferric fluoride (FeF₃) by the chemical reaction between the Fe₃O₄ and hydrofluoric acid/nitric acid mixture solution during the preparation process. To overcome these challenge, we suggest the approach to combine the phosphorus (P)-doped (NH₄)₂SiF₆, as a novel fluorescent materials, with Fe₃O₄ without the formation of FeF₃. In this presentation, we report the fluorescence color and chemical composition of the magnetofluorescent microparticles. The P-doped (NH₄)₂SiF₆ microparticles were linked with the Fe₃O₄ using ultrasound cleaner. To vary the fluorescent color of the P-doped (NH₄)₂SiF₆ microparticles, they were immediately treated in vacuum or in atmospheric environments. The fluorescence properties and chemical composition of the obtained microparticles were investigated by using photoluminescence (PL) and X-ray photoelectron spectroscopy (XPS) measurements. The fluorescent color varied from yellow to green light due to the oxidation of the P-doped (NH₄)₂SiF₆ in atmospheric environment. Furthermore, the magnetic component in the microparticles was composed of only Fe₃O₄ without the formation of FeF₃. Therefore, we suggest that the particles become a highly effective tool of diagnosis and treatment of cancer cells as a novel magnetofluorescent materials.

NM-WeP11 Nonideality in Atomic Layer Deposition and Its Implication in Efficient Electrolysis, Changdeuck Bae, T.A. Ho, H. Shin, Sungkyunkwan University, Korea, Republic of Korea

We describe the spontaneous formation of a composite chalcogenide materials that consist of two-dimensional (2-D) materials dispersed in bulk and their unusual charge transport properties for application in hydrogen evolution reactions (HERs). When MoS₂ as a representative 2-D material is atomic layer deposition-deposited on transition metals such as Cu in a controlled manner, the sulfidation reactions also occur with the metal. This process results in remarkably unique structures, i.e., bulk layered heterojunctions (BLHJs) of Cu-Mo-S that contain MoS₂ flakes inside, which are uniformly dispersed in the Cu₂S matrix. The resulting structures were expected to induce asymmetric charge transfer via layered frameworks and tested as electrocatalysts for HERs. Upon suitable thermal treatments, the BLHJ surfaces exhibited the efficient HER performance of approximately 10 mA/cm² at a potential of as low as -0.1 V versus a reversible hydrogen electrode (RHE). The Tafel slope was approximately 30 to 40 mV/dec. The present strategy was further generalized by demonstrating the formation of BLHJs on other transition metals such as Ni. The resulting BLHJs of Ni-Mo-S also showed the remarkable HER performance and the stable

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operation over 10 days without using Pt counter electrodes by eliminating any possible issues on the Pt contamination.

NM-WeP12 Stretchable Temperature Sensor Based on Elastomeric rGO/PU Nanocomposite Fiber, *Tran Quang Trung, N.-E. Lee*, Sungkyunkwan University, Republic of Korea

Developing fiber-based sensors and integrating them with textile to develop smart textile smart clothes, and smart gloves for monitoring physiological and biological signals of human body are promising for next generation of wearable electronic platform. In recent, there have been many efforts on development of stretchable temperature sensors. However, they still presented some limitations such as requirement of complicated fabrication process, high cost, low yield, and unstability in output performances under mechanical deformation. Herein, we proposed a stretchable temperature sensor with simple fabrication process based on elastomeric rGO/PU nanocomposite fiber. By incorporating high thermal-responsive material (reduced graphene oxide (rGO)) into outstanding mechanically stretchable elastomer polymer (polyurethane (PU)), we generated a free-standing rGO/PU elastomeric composite fiber with high thermal responsivity and good mechanical deformation via a simple fiber spinning method. The fiber with outstanding mechanical deformation helps it easily moulded in serpentine shape and encapsulated by polydimethylsiloxane (PDMS) to generate a free-standing stretchable temperature sensors. Fabricated device presented high responsivity, stretchability, and stability in sensitivity under applied stretching. The free-standing stretchable temperature sensor can be embedded on PDMS substrate, sewn on stretchable fabric and bandage, and laminated on human body to monitor the temperature changes of skin during doing exercise. These results demonstrate that the free-standing, fiber-based, stretchable temperature sensor has great potential for development of smart textile for personal healthcare.

NM-WeP14 Au Nanoparticle Decorated rGO/MoS₂ Sandwich Catalyst for Photodegradation, *Jyh-Ming Ting*, National Cheng Kung University, Republic of China

With the development of industry and the rapidly innovative technology progress, various pollution issues have thus occurred. For example, management of wastewater is one of the critical issues. Therefore, waste water purification by semiconductor or semiconductor composite photocatalyst has been attracting much attention. In the study, we have investigated a novel catalyst for use as photocatalyst. The photocatalyst is a novel visible light response Au nanoparticle decorated rGO/MoS₂ material. The synthesis of such photocatalyst involves the use of a supercritical fluid carbon dioxide process. The processing conditions were studied to obtain desired characteristics of the photocatalyst. The obtained photocatalyst was for the degradation of MB.

NM-WeP15 Surface Modification of CFRP by CNT-Doped Buckypapers, *Bartłomiej Przybyszewski, K. Dydek, P. Latko-Duralek, A. Boczkowska*, Warsaw University of Technology, Poland

One of the latest trends in the development of new materials is to improve the electrical and thermal conductivity of Carbon Fiber Reinforced Polymers (CFRP). Instead of metallic meshes, lightweight structures are searching as more promising candidates that decrease the total weight of composite laminates and are electrically conductive in order to be able to provide electromagnetic shielding.

Thin sheets made from an aggregate of carbon nanotubes (so called buckypapers) are novel CNT-based materials which mainly consist of carbon nanotubes that are bonded together by thermal treatment. They can improve the electrical conductivity of CFRP composites due to the presence of carbon nanotubes (CNTs), which are known to be highly conductive nanoparticles.

In order to investigate the effect of surface modification of CFRP by CNT-doped buckypapers on the electrical and thermal conductivity of CFRP, two types of laminates were fabricated with different buckypaper types. All panels were made from carbon prepregs using a vacuum bag (Out of Autoclave method). Electrical conductivity was measured in three directions of the fabricated panels. The differences in the results were explained by microscopic observations. In addition, the effect of CNT-doped buckypapers on the thermal properties of the panels was analyzed.

NM-WeP16 Characterizing the Quality of Molten Al Alloys with Hydrogen, Porosity and Bifilm Content, *H. Jang, P. Youn, H. Kang, G. Lee, J.B. Jeon, J. Park, E. Kim, Sunmi Shin*, Korea Institute of Industrial Technology, Korea

In response to the recent strengthened environmental regulations, high strength aluminum alloys have been actively studied as a substitute for

steel material for light weight of vehicle. In particular, Al-Mg alloy is a typical high-strength aluminum alloy, and it is used as a structural material with excellent strength and deformation resistance. Mg alloying element induces solid solution hardening and precipitation hardening depending on coexistence of Si and Zn. On the other hand, Mg element weakens the fluidity of the molten alloy and increases the amount of inclusions due to the strong bonding force with oxygen. In order to reduce the internal defects and improve the mechanical properties of Al-Mg alloys, the quality of molten Al-Mg alloys should be precisely controlled. In this study, the melt quality of Al-Mg alloys was characterized by measuring hydrogen, porosity and bifilm content and the effect of Mg content and melting process parameters on the melt quality was discussed.

NM-WeP17 Molecular Confinement on Nanostructured Polymer Surfaces, *Sara Heedy, A.F. Yee*, University of California, Irvine

Polymers have a characteristic size (lamella thickness, molecular orientation, etc.) which may depend on processing, especially when fabricated into structures that range from 10's to 100's of nm. These dimensions may be important in certain properties such as electrical or thermal conductivity. These properties will be strongly affected when fabricated polymer nanostructures have dimensions comparable to the critical length scale of physical phenomena (unconstrained radius of gyration, mean free path of charge carriers, mass transport, etc.). Enhanced mechanical, optical, and electrical properties of nanostructures, including arrays of nanopillars and nanolines less than 1 μm tall, have been well documented. Scientists and engineers who desire to control such properties are often unable to characterize and predict the nanoscale surface chemistry change due to surface topography. This research aims to significantly update the current knowledge of nanoscale surface chemistry, allowing the alteration of physical properties in a defined and predictable manner to obtain unique bio-electronic interfaces. Here, we aim to both quantify and control interface chemistry and molecular confinement to allow researchers the ability to alter surface chemical composition on a given polymer substrate in a methodical and predictable manner.

We fabricated nanostructures using nanoimprint lithography on the synthetic polymer poly(methyl methacrylate) (PMMA), which is often used for biomedical devices. A consequence of such a process is confinement induced reordering of polymer chains which is strongly affected by the mold geometry, mold surface properties, and imprinting process variables. Using thermal imprinting, and the combined topographical and nanoscale chemical mapping of photoinduced force microscopy (PiFM) (Nowak et al. Sci. Adv. 2016), we found that nanopillars (100-700 nm range) confine functional groups differently depending on the pattern geometry used. These findings are very surprising, and suggest that surface chemistry, as well as nanoscale phenomena, can be controlled for use in adhesion and bio-electronic interfaces.

NM-WeP18 Non-volatile Memory Based on Negative Capacitance and Photovoltaic Effect, *Kai-Wen Chen, S.-Y. Chen, Y.-C. Tseng, S.-J. Chang*, National Chiao Tung University, Republic of China

The investigation for solid-state non-volatile memory has triggered great research into new materials combination. Here we demonstrate that the significant photovoltaic effect of the multiferroic heterostructure. This work GdFe_{0.8}Ni_{0.2}O₃/SrTiO₃ (GFNO/STO) ferroelectric capacitors have been successfully fabricated on an Nb-doped SrTiO₃ substrate, in which the GFNO and STO film were prepared by magnetron sputtering. When GFNO layer was combined with an STO layer, the total capacitance appeared increased, which suggested the negative value of the ferroelectric capacitance. In addition, the amplified photocurrent was induced by positive poling which led to polarization pointing down and vice versa. The direction of photocurrent could be reproducibly switched along with the polarization flips. Using x-ray absorption spectroscopy (XAS) with in-situ electrical characterization, we investigated the poling dependence of transport properties at an interface of a GFNO/STO devices. We found that electrons were pushed upward or downward by dipole moment inside GFNO thin film with the occurrence of polarization flipping which decreased the rate of recombination of electron-hole pairs. In addition, photoelectron spectroscopy (PES) indicated that oxygen vacancies accumulated mainly at one side of GFNO with poling dependence and motivated the change of Schottky barrier height at the interface. With the combination of modern x-ray technique and photocurrent measurement, our results pave a way for ferroelectric electro-optic memory by manipulation of multiferroic interfaces. This work was financially supported by the "Center for the Semiconductor Technology Research" from The Featured Areas Research Center Program within the framework of the

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