**Highlights of the Faraday Discussion on Nanoparticle Synthesis and Assembly, Argonne, USA, April 2015**

Xiao-Min Lin, Yugang Sun, Elena V. Shevchenko, Subramanian K. R. S. Sankaranarayanan, Daniela John, Igor Fedin, Fernando Bresme, Helmuth Möhwald, Philip Moriarty, Christopher M. Sorensen and Bruce M. Law

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Cite this: DOI: 10.1039/c5cc90369f

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The Royal Society of Chemistry Faraday Discussion (FD) conferences have a history of nearly a hundred years, and yet there have been only two previous meetings held in the United States.¹ This makes the third meeting, held at Argonne National Laboratory (ANL), USA on April 20–22, 2015 (Fig. 1), particularly special. The conference focused on the topic of nanoparticle synthesis and assembly, an exciting research area that has seen many remarkable scientific breakthroughs during the past two decades. It was co-sponsored by Kansas State University (KSU), ANL's Center for Nanoscale Materials (CNM) and Cogent Inc., with 125 attendees coming from 23 countries.

The meeting officially kicked off on Monday, April 20, with Argonne Deputy Director **Alfred P. Sattelberger** and conference scientific committee chair **Bruce**

M. Law from KSU. Following tradition, the Faraday Discussion Spiers Memorial Award is given to an opening speaker who has made outstanding contributions to a particular Faraday Discussion topic.² This year's Award was given to **Paul Alivisatos** from the University of California at Berkeley, ~~the opening speaker for this FD conference,~~ for his groundbreaking contributions towards understanding fundamental physical chemistry properties of nanocrystals. In his opening comment, Paul quoted **Ernest Rutherford's** famous statement: "All science is either physics or stamp collecting." He presented an interesting question: whether our field needs to go beyond the stamp collecting process and dig deeper in order to understand the intrinsic mechanism of nanoparticle synthesis and

assembly. He presented a glimpse of the exciting science that can be achieved using recently developed *in situ* transmission electron microscopy techniques. One example he gave was on studying Pt nanoparticles in a graphene liquid cell. Using the thinnest membrane to encapsulate a tiny liquid droplet, Paul and his colleagues have demonstrated that it is possible to directly image a growing nanoparticle in a real environment ~~with atomic resolution.~~ This study showed particles growing *via* coalescence and orientated attachment events, in addition to the monomer nucleation process.³ On the topic of nanoparticle self-assembly, Paul gave an example of tracking the trajectory of Au nanorods, from which the interparticle potential can be directly measured. By adjusting the ionic

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Fig. 1 Center for Nanoscale Materials (top) and Advanced Photon Source (bottom), Argonne National Laboratory, U.S.A., was the site of the FD on Nanoparticle Synthesis and Assembly, 2015.

strength of the solution, the range and strength of the interparticle potential can be tuned, which can create a highly anisotropic potential and drive the end-to-end nanorod assembly.⁴ The exciting discussion following Paul's opening remarks set the tone for the entire conference. Questions were raised on whether the electron beam itself has an impact on the phenomena observed in such *in situ* experiments, and whether sample heating by the electron beam is a problem. Paul replied that the temperature increase for nanoparticles in a liquid environment is not significant, and by studying the flux-dependence of the process, the issue of beam induced effects can be distinguished.

The control over morphology and composition at the nanoscale has remained a hot topic over the past decade. Colloidal synthesis is a great way to create a broad range of nanostructures with controlled compositions and shapes. ~~Understanding the mechanism of nucleation and growth of these structures is a central task in nanoscience.~~ The first session on Monday afternoon focused on new routes to control nanoparticle synthesis, and was chaired by **Elena V. Shevchenko** (ANL). **Christophe Petit** (Université Pierre et Marie Curie)

investigated experimentally and theoretically the role of interactions of small adsorbed molecules on the shape and composition of platinum and platinum-based nanoalloys.⁵ He followed the growth evolution of such nanoparticles *in situ* using small-angle X-ray scattering, and confirmed that the presence of hydrogen atoms adsorbed at the platinum surface strongly modifies the kinetics of the nanoparticle growth process. This phenomenon has also been predicted by density function theory (DFT) calculations. **Peter Schurtenberger** (Lund University) described a new route to assemble colloidal molecules using polymer microgels.⁶ His approach is based upon the ability of microgels to be adsorbed at the oil/water interface, resulting in anchoring of the microgels by the tiny oil droplets. Different types of microgels can be assembled from their mixtures allowing the synthesis of patchy particles. Capillary forces are found to be the driving force for such assemblies at the early stages of their formation. The charge and size of the microgels can be used to vary the interaction potentials to create tunable binding sites.

Following a brief afternoon coffee and tea break, in which attendees had their first chance to mingle, the second

session chaired by KSU's **Christopher M. Sorensen** proceeded with great excitement. Biological assembly of magnetite nanoparticles (magnetosomes) into nearly perfect linear structures allowed magnetotactic bacteria to sense the Earth's magnetic field. **Damien Fairve** (Max Planck Institute of Colloids and Interfaces) discussed how two proteins, Mam-J and Mam-K, facilitated the assembly of magnetosomes into linear arrays, thus overcoming the lower energy configuration of a closed loop.⁸ Using high-resolution optical microscopy, they showed MamK filaments were widely dispersed within the cytoplasm of the cell and therefore their localization was not limited to the magnetosome chain. This work serves as an important example of nanoparticle assembly *via* biomimicry (Fig. 2).

Given their huge specific surface area, nanoparticles are expected to be chemically very reactive. However, one also expects that a stabilizing ligand shell on the nanoparticles could buffer against reactivity. In this regard, **Christina Graf** (Freie Universität) presented a thorough study of the effects of halide ions on the stability and morphology of gold nanoparticles in aqueous solution.⁹ The gold nanoparticles were *ca.* 5 to 6 nm in diameter and ligated with mono-, di- and tri-thiolated polyethylene glycol (PEG). Chloride solutions were stable, but bromide led to a general coarsening of the colloid with time, more so at modestly elevated temperatures. Iodide solutions showed significant changes, readily apparent in both UV-Vis spectroscopy and TEM, which in some situations showed larger prismatic structures. These results were explained in terms of the hard/soft acid/base concept with gold being a soft acid, and the halides being bases with increasing softness from chloride to iodide. It was also observed that the stability against halide attack increased with the valency of the PEG thiols. These results are important not only with regard to colloidal stability but also for particle shape and size manipulation.

Dmitri Talapin (University of Chicago) gave an invited discussion on the topic of using inorganic ligands, such as metal

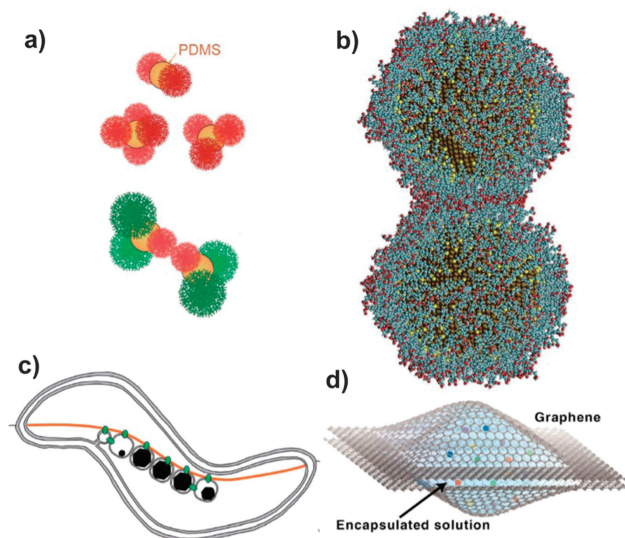


Fig. 2 New trends in nanoparticle synthesis presented at the FD meeting. (a) Control of the interaction potential to achieve designed patchiness of particle aggregates.⁶ (b) Molecular dynamics simulation to understand the configuration of ligand on the nanoparticle surface.⁷ (c) Biomimetic approach to control nanoparticle synthesis and assembly.⁸ (d) *In situ* TEM technique to understand nanoparticle growth and assembly.³

chalcogenide complexes, oxoanions/oxometallates, and halides/pseudohalides/halometallates, to passivate the nanoparticle surface.¹⁰ His group has over the years demonstrated that, with these modifications, charge mobility in nanoparticle superlattices can be greatly enhanced.¹¹ Semiconductor nanocrystals capped with such inorganic ligands show great promise in the fabrication of solution-processed electronic devices such as field-effect transistors (FETs), photodetectors, light-emitting diodes (LEDs), and solar cells.

Monday afternoon's poster session and wine reception were sponsored by Cogent Skills, Inc. There were 70 contributed posters focusing on a variety of research topics, among which many exciting new scientific developments were presented. As a result, the scientific committee had a hard time to choose the student poster award. Ultimately, **Igor Fedin** (University of Chicago) and **Daniela John** (Fraunhofer Institute for Applied Polymer Research, Potsdam-Golm, Germany) were chosen for their exciting science, as well as great presentation style. Igor used potentiometry to study the interaction of negatively charged inorganic ions, such as sulfide (S^{2-}), with the surfaces of the semiconductor nanocrystals cadmium selenide (CdSe) and indium phosphide (InP).¹² With the potentiometric probe immersed in the solution, he showed that it is possible to controllably grow multiple layers of CdS onto CdSe nanocrystals. Potentiometry thus provides real-time feedback during the synthesis of core-shell nanocrystals where this technique saves material and reduces chemical waste from purification. Daniela's research focused on the alignment of cyclodextrin-covered particles. Cyclodextrin has a ring-like structure built on glycoside molecules and can form supramolecular interactions with other, smaller molecules (host-guest interaction). She used wrinkled substrates as a template for the alignment of particles directly from solution. By controlling parameters, such as concentration, pH, particle size, and wrinkle geometry, excellent alignment can be achieved *via* dip-coating or drop casting.

On the morning of April 21, **Yugang Sun** (ANL) chaired the third session that focused on nanoparticle synthesis. The two fascinating talks in this session both focused on patchy particle colloids. Inspired by the self-assembly behaviour of telechelic star polymers, **Emanuela Bianchi** (University of Vienna) introduced a class of patchy particles, *i.e.*, soft and flexible patchy colloids (SFPCs), with a soft, repulsive core on which a fixed number of soft, attractive patches are attached.¹³ In each SFPC particle, the directional bonding, soft interactions, and incessant patch rearrangements are complemented with energy penalties associated with the mobility of the patches, reversible modifications of the patch number/size and the possible formation of more than two bonds per patch. Monte Carlo simulations have been explored to predict the bulk behaviour of the fluid phase at a fixed density with a particular focus on the impact of the patch flexibility on the percolation threshold in the SFPC systems. Enthalpic patches (*i.e.*, polystyrene nodules) and entropic patches (*i.e.*, dimples) on silica particles can be synthesized through dedicated colloidal chemistry. **Etienne Duguet** (CNRS, University of Bordeaux) demonstrated the use of silica particles as seeds to promote the heterogeneous nucleation and growth of polystyrene nodules through an emulsion polymerization reaction.¹⁴ The number of polystyrene patches on each silica particle is determined by the size of both the silica particle and the polystyrene nodules. Selectively overgrowing the silica particles in the silica/polystyrene binary multipods followed by selectively dissolving the polystyrene nodules leads to the formation of silica particles with dimple patches.

The first session focussing on the self-assembly process was chaired by **Xiao-Min Lin** (ANL). Some surprising results were presented by **Brian Korgel** (University of Texas) on nanoparticle superlattices. Normal atomic crystals undergo a thermally induced melting transition where crystals melt into a disordered liquid state at high temperature. Brian showed that in body-centred cubic (bcc) superlattices of small nanoparticles, a

mild heating can induce better ordering in the superlattice structure.¹⁸ More astonishingly, this disorder-to-order transition is reversible with temperature. A hypothetical model of ligand melting and solidification was used to interpret these results. Very few studies have so far been done on understanding the stability of colloids involving dissolving nanoparticles into ionic liquids. **Régine Perzynski** (Université Pierre et Marie Curie) reported a detailed study on maghemite nanoparticles in ethylammonium nitrate.¹⁹ Their work found that the particle size, the nature of the initial counterion in water, and the amount of water in the ionic liquid can all influence the interparticle potential and modify the phase diagram of the system. Delivered *via* an online web conferencing system, **Gunadhor S. Okram** (UGC-DAE Consortium for Scientific Research) described their study on nickel nanoparticles using different ligands, including trioctylphosphine (TOP), triphenylphosphine (TPP) and oleylamine (OA), and showed that different ligands affect both the monodispersity of the sample as well as their ability to order into superlattices.²⁰

Surfaces are frequently used to assemble two-dimensional nanoparticle arrays, usually *via* either evaporative or templated assembly. In Tuesday afternoon's session on drying mediated self-assembly processes, chaired by **Bruce Law** (KSU), **Casper Kunstmann-Olsen** (University of Liverpool) *et al.* used environmental scanning electron microscopy (ESEM) to image *in situ* various structures formed during the evaporation of aqueous nanoparticle droplets on various wetting/non-wetting substrates, in which the water evaporation and recondensation rate was controlled.²¹ Their most surprising results occurred in the presence of dissolved salt in the nanoparticle solution. The dissolved salt strongly influences the surface distribution of precipitated nanoparticles. **Lucio Isa** (ETH Zurich) gave a fascinating talk on capillary assembly of nanoparticles using templated rectangular traps.²² As the liquid evaporates, nanoparticles accumulate in the vicinity of the liquid meniscus/template contact line and fill the traps with 0, 1 or 2 particles, where

the number of particles per trap depends upon the relative trap depth to particle size as well as the surface tension of the evaporating liquid. A similar strategy was employed by **Andreas Fery** (University of Bayreuth) *et al.* to assemble nanorods into linear arrays through dip coating on a wrinkled substrate.¹⁵ Single, double or triple nanorod lines were formed, depending upon the wrinkle amplitude. Single nanorod lines exhibit very high fidelity in terms of nanorod orientation and gap size between rods. The authors used capillary assembly to transfer single nanorod lines from the hydrophobic wrinkled-substrate onto a hydrophilic indium tin oxide (ITO) substrate. A water droplet between the two substrates, together with light pressure applied to the wrinkled substrate, mediates this capillary assembly.

After the tea and coffee break, the second session concerning nanoparticle assembly was chaired by **Fernando Bresme** (Imperial College). The main part of the session focused on the experimental and modelling aspects of the mechanical properties of nanoparticle (NP) assemblies. **Yifan Wang** (University of Chicago) *et al.* explained how alkanethiol-coated nanoparticles can form monolayers with remarkable

mechanical strength that can be suspended as free-standing nanoparticle membranes (NPM) over micron-sized holes.¹⁷ The analysis of the fracture behaviour of these membranes on a PDMS stamp provides important insights into the distribution of local bond strengths in the bilayers. The membranes can be rolled up using electron beam irradiation, driven by ligand distribution asymmetry and electron beam induced cross-linking, hence providing a route for practical applications (Fig. 3). **Michael Salerno** (Sandia National Laboratory) discussed large-scale atomistic simulations of nanoparticle arrays.⁷ This work provides valuable insight into the microscopic origin of the mechanical strength of NPM, which, according to the simulations, is determined mainly by ligand–ligand interactions. Interestingly, computer simulations indicate that strong ligand interdigitation might not be essential in achieving large mechanical strengths. **Subramanian Sankaranarayanan** (ANL) gave an invited discussion remark. He discussed coarse-grained molecular dynamics simulations of nanoparticles. Coarse-grained simulations have achieved a high degree of sophistication, making it possible to simulate large NP

arrays for long times (10^{-6} s) and length scales (10^{-7} m). These simulations provide surprising insight into the role of ligand coverage in determining the NP ligand shell asymmetry.²³ Such asymmetry develops during membrane assembly at the air–water interface, and ultimately influences the mechanical response and unidirectional bending of NPM. **Toshiharu Teranishi** (Kyoto University) discussed the optical properties of copper chalcogenide nanodisks.²⁴ His contribution highlighted the relevance of the nanoparticle in-plane oscillation mode in defining plasmon resonance peaks in the near-infrared, a frequency domain that is relevant in photothermal therapy applications. Ligand exchange of oleylamine and 1-dodecanethiol provides a versatile route to tune the nanodisk composition and to synthesize nanoparticle arrays with distinctive self-assembly behaviour.

Right before the dinner, the Center for Nanoscale Materials User Office organized a facility tour and many conference attendees participated. As one of the Department of Energy user facilities for nanoscience, CNM offers many cutting-edge facilities to study nanomaterials, including a state of the art clean room facility, an advanced scanning tunneling microscopy facility, chromatic aberration corrected TEM, and an optical characterization facility. A premier instrument is CNM's unique hard X-ray nanoprobe beam line at the Advanced Photon Source, which provides scanning fluorescence, scanning diffraction, full-field transmission and tomographic imaging capabilities with a spatial resolution of 30 nm.²⁵ Recently, synchrotron X-ray scanning tunneling microscopy (SXSTM) was added as another way to provide chemical imaging at high spatial resolution.²⁶

A long day of exciting talks and discussions created a friendly atmosphere among the conference attendees, which permeated throughout the evening dinner that followed. The fun culminated in a hilarious rendition of the Loving Cup Ceremony. Although the original silver cup didn't make the trip across the Atlantic, all the attendees still enthusiastically followed the long tradition of Faraday

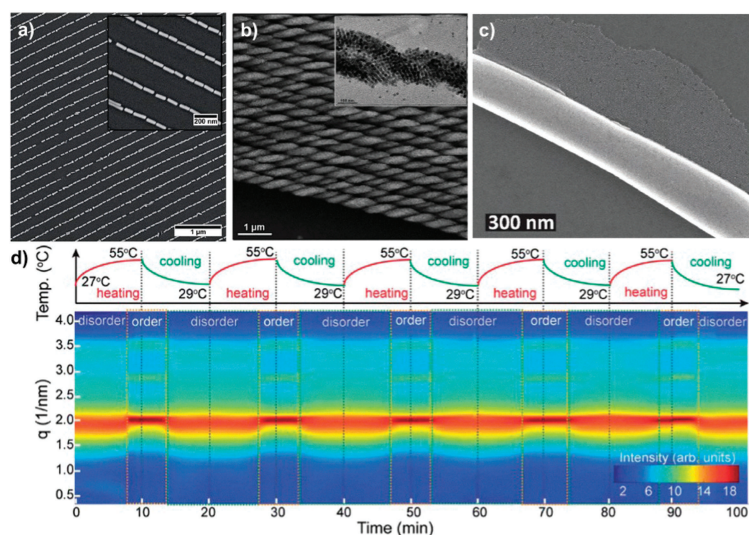


Fig. 3 New advances in nanoparticle assembly presented at the FD conference at Argonne. (a) Optically anisotropic surface created by drying-mediated assembly of nanoparticles on a wrinkled substrate.¹⁵ (b) Helical superstructure of Fe₃O₄ nanocubes assembled under an applied magnetic field.¹⁶ (c) Folding of a 2D nanoparticle membrane sheet into a tubular structure.¹⁷ (d) Reversible order–disorder transition in a bcc superlattice made of 1.67 nm octadecanethiol-ligated Au nanocrystals monitored by GISAXS.¹⁸

Discussions with a toast of “*In piam memoriam of GS Marlow and Angela & Tony Fish.*” Finally, glasses were raised for Professor Bruce Law who conceived of this Discussion and led the conference organization.

The last day of the meeting, on April 22, had two sessions that captivated most attendees’ attention, both devoted to field-assisted self-assembly processes. The ability to direct the assembly of colloidal nanoparticles *via* application of external stimuli, such as electric and magnetic fields into one, two or three-dimensional superstructures is of fundamental and practical interest. The first session, chaired by **Helmuth Möhwald** (Max Planck Institute of Colloids and Interfaces), started with a very promising approach towards the application of nanotechnology by **Nicholas Kotov** (University of Michigan). He demonstrated a very elegant way to prepare a rough surface of a cheap metal, such as copper, by electric field-assisted deposition of nanoparticles.²⁷ Compared to conventional electrochemical deposition of ions, nanoparticle deposition requires current more than an order of magnitude lower, and these low currents obviously serve to fabricate conductive junctions between the particles. This good connectivity, which was also demonstrated by high resolution microscopy, allows for fast charge transport. Thus, a low-energy consuming method was presented to develop capacitors with at this stage

capacity nearly a factor of 2 greater compared to conventional methods, and the high surface area electrodes were also promising for Li batteries. In addition, Nicholas presented chiral arrangements of nanoparticles created using circularly polarized light.²⁸ Following a very similar concept, **Rafal Klajn** (Weizmann Institute of Science) and **Petr Král** (University of Illinois at Chicago) presented an experimental and theoretical study of the chiral assembly of cubic magnetic nanoparticles.¹⁶ In these crystalline nanoparticles, there is an angle between the magnetic easy axis and the crystallographic axis. The magnetic interactions hence form an angle with respect to the short range van der Waals interactions and this leads to a chiral structure. This structure can be manipulated by a magnetic field, and there is remarkable agreement between theory and experiment. The local interactions can in addition be manipulated by adsorbed ligands, and here a photosensitive ligand was demonstrated to yield photoswitchable structures. As the chirality is spontaneous, both enantiomers were produced in equal proportion. In the following discussions in relation to chirality and the origin of life, questions on chiral selection were raised. It was suggested this could occur *via* chiral impurities or *via* adsorption on chiral structures such as mineral surfaces.

In the second session, chaired by ANL’s **Subramanian Sankaranarayanan**,

Orin Velev (North Carolina State University) and co-workers presented one such example wherein a magnetic field is used to drive the assembly of lipid-coated iron oxide nanoparticles into microfilaments that display remarkable flexibility and self-healing properties.²⁹ The magnetophoretic and the inter-particle dipole-dipole interaction are identified as the primary attractive forces driving the assembly of nanoparticles in the presence of a uniform magnetic field. The capillary bridges between the lipid layers balance the electrostatic repulsion between the particles and bind the nanoparticles within the microfilaments. Furthermore, the response of this assembled superstructure to changes in the direction of the magnetic field was also presented. The filaments were shown to be reconfigurable into discrete bundles in a 2-D rotating magnetic field. Theoretical models can further help in improving our understanding of the nature of the inter-particle interactions and establish design rules for tuning the emergent magnetic properties of such self-assembled superstructures. Towards this end, **Suvojit Ghosh** (McMaster University) simulated the dynamics of interacting magnetic moments in the assembled superstructures by applying the stochastic Landau–Lifshitz–Gilbert equation.³⁰ A two-particle model was used to demonstrate that superparamagnetic nanoparticles can be organized into ferromagnetic structures with substantial residual magnetization. The nature and type of the nanoparticles, as well as their size, were shown to have a profound effect on the resulting magnetic properties.

An exciting three-day Faraday Discussion came to an end with inspiring closing remarks given by **David Schiffrin** (University of Liverpool). David started his remarks by recalling a bit of history in **Michael Faraday’s** research notebook; he pointed out Faraday’s extraordinary instinct and enthusiasm when he made a new discovery. David encouraged the attendees to communicate this kind of excitement to the young generation of scientists, encouraging them to find their own success in research. This Faraday Discussion offered exactly this kind



Fig. 4 Snapshots from the FD meeting. (a) Paul Alivisatos receives the Spiers Memorial Award from conference chair Bruce Law. (b) David Schiffrin delivers inspiring closing remarks. (c) Discussions at the poster session. (d) Scene from the main conference room.

of stimulating and exciting environment for young scientists to grow, and also showed that nanoparticle synthesis and assembly is a very active research area, with new and exciting discoveries to be made in the near future (Fig. 4).

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