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Nanoscale Architectures for Smart Bio-Interfaces: Advances and Challenges

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1. Introduction

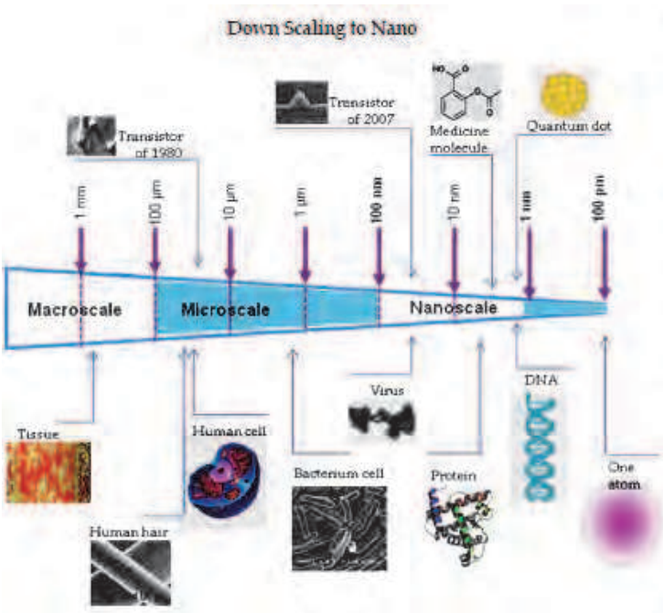
"*At the nano level atoms do not belong to any field of science*", professor Chad Mirkin explained the need to unlock our minds and transform our attitudes, as we continue to live the adventure of nano science and technology. This ably conveys both the uniqueness and diversity of nanotechnology, while stressing the preparation required by those aspiring to it (Papapzoglou & Parthasarathy, 2007). Clearly, the structuring of substance at the molecular scale extend across the entire spectrum of scientific knowledge counting physics, chemistry, medicine, or engineering.

However, why should anyone care about *nanotechnology* or even *nanofabrication*? If we take a step back, we find that the *microfabrication* techniques, such as the conventional lithography, deposition, or etching have enabled micromachining of architectures down to sub-micrometer dimensions, (e.g., 400-900 nm). These techniques have attained an adequate level of maturity and can be found already incorporated commercial MEMS products, like pressure microsensors, micro-accelerometers, or micro-gyroscopes (Arshak, 2005; Cook-Chennault, 2008; Guo, 2009; Liu, 2007; Pal, 2006; Tsai, 2007). Other research-grade, sensitive micro(bio)interfaces (Amatore et al, 2006; Asher et al, 2002; Avramescu et al, 2002 ; Bitziou et al, 2010; Cosnier, 2000; Deo et al, 2003; Grayson, 2004; Huber et al, 2006; Jungblut et al, 2009; Knoll et al, 2006; Lee et al, 2009; Marcon et al, 2010; Peteu et al, 2007; Szunerits & Walt, 2002; Vasilescu et al, 2003 ; Ziaie, 2004a).

More recently, nano-size structures have attracted a colossal interest, due in part to their unique electrical, magnetic, optical, thermal, and mechanical properties. Clearly, once properly developed, these *nano* architectures are expected to lead to a range of electronic,

photonic, sensing or actuating devices with superior cost-benefit performances, compared with their *macro* and *micro* counterparts. Herein, the focus is on advances and challenges in nanofabrication of smart bio-interfaces. While staying true to such fascinating thoughts, our efforts aim to achieve the control at molecular scale. By building block-by-block the new nano-architectures, one will not only advance scientific knowledge, but also will design and develop the future nano-parts for the next-generation hybrid smart micro-devices. These will integrate multi-level hierarchically linked nano-parts with electrical, optical, chemical, biological functions. Several nanofabrication methods are more mature, others are currently intensely investigated however these are not *per se* the subject of this chapter. More details can be found in well-documented, recent books, chapters or reviews (Bhushan, 2004; Cui, 2008; Kim et al, 2005; Klauser et al, 2010; Kummar et al, 2005; Mirkin & Rogers, 2001; Wang, Mirkin & Park. 2009; Ziaie, 2004b).

The *nanofabrication* implies making artifacts whose scale is in the nano domain, *1 to 100 nanometers*. As a reminder, 1 nm is one-millionth of 1 mm, 1 nm = 10^{-9} mm. To setup stage for nano, **Figure 1** charts a comparison between nano-scale and several familiar components: one human hair, the human cell, an 1980-old transistor, a bacterium, a virus, DNA, etc.



Direct and Indirect Nanofabrication	
Direct	**Resolution**
Optical lithography	30 nm
Electron beam lithography	10 nm
Nano-imprint lithography	10 nm
Scanning Probe lithography	20 nm
Reactive ion etching	50 nm
Metal lift-off	10 nm
Focused ion beam	30 nm
Indirect	
Sidewall lithography	50 nm
Dimensional substract.-addition	50 nm
Self-assembly, DNA directed	*20 nm*
Multi-step processing	30 nm
High resolution nano-patterning	2 nm

Fig. 1. The nanofabrication methods /resolution, compared with manmade *vs.* natural micro-nano worlds.

Among nanofabrication methods listed in Figure 1, self-assembly often appears as most promising, due to its low cost and remarkable ability to produce nanostructures at different length scales. Consequently, IBM and Cal Tech teamed up to build *DNA origami* at the sub-22 nm scale, this nano-circuitry using DNA-directed self-assembly, by dry etching the SiO_2 with e-beam lithography, to create sites for the DNA to attach & self organize. Thus, DNA becomes a scaffold for nanotubes to self-assemble into sub-22 nm circuits (e.g., Aldaye & Sleiman, 2009; Nanofabrication , 2011 course web).

The strongest driving force for nanofabrication remains perhaps the production of increasingly smaller electronic components, keeping pace with *Moore's Law* forecasting the

doubling of IC device density every 18 months (Freebody, 2011; Kahng, 2010). Furthermore, depending on specific applications, additional vectors could be desirable, including: complexity of structures, feature density, and materials generality. In addition, the fidelity/accuracy and scalability (parallel, simple, and cost-effective processing) are also keys to develop new and useful nanofabrication technologies (Wang, Mirkin & Park, 2009). One valued consequence of this drive is providing researchers with new tools and nano-materials to address rewarding topics in materials science and engineering, energy, life sciences, healthcare and more.

Nanofabrication offers two approaches, *top-down* and *bottom-up*. The *top-down* nanofabrication constructs objects from larger entities by removing material, while the *bottom-up* nanofabrication builds devices structured devices *via* the assembly of their molecular parts. While high-resolution lithography methods, like e-beam, can be employed to fabricate nano-size structures, their serial nature and or cost preclude a widespread application. This has forced investigators to explore alternative and potentially superior techniques such as self-assembly or nanoimprint lithography.

This chapter will critically review several specific nanoscale architectures for smart interfaces, selected as being under intense investigation and with exciting advances reported (Ali et al, 2008, 2010; Baca et al, 2011; Chi et al., 2005; Ishihara & Takai, 2009; Iwamoto, Kaneto & Mashiko, 2003; Kim et al., 2009; Knoll et al., 2008; Shvedova et al, 2010; Niedziolka-Jonsson, 2010; You et al., 2006, 2007). The following nano interfaces will be appraised:

1. Nano scale modeling and simulation, enabling nano-fabrication by-design, or "rationally designed materials";
2. Hybrid organic-inorganic nanomaterials, specially conductive polymer hybrids for enhanced sensing and actuation;
3. Nanoplasmonic methods and structures with focus on lamellar plasmonic nano-interfaces for optical sensors;
4. Nanoelectronics and more specifically the controlled molecular functional architectures for thin film transistors.

2. Modeling and simulation of nano architectures for rationally designed materials

2.1 The need for rational nanomaterials design

The investment in nanoscience has been seen by many countries as building a new chance for the future. The United States has one such team effort, the *Chemical Vision 2020* partnership, a chemical industries lead effort (GE, Dow, Intel, DuPont, Honeywell, Ciba, Rohm and Haas) to accelerate innovation and technology development, to introduce a host of innovative products to revive and energize the economy, solving major societal problems and creating new businesses. There is a resilient confidence that Nanofabrication will catalyze the new manufacturing (www.chemicalvision2020.org).

The traditional, typical methods to discover and develop new nanomaterials, today, are still based on somewhat focused *experimentation* and *scientific inspiration*, rather than on *rigorous engineering design*. A new strategy to achieve rational nanomaterials design (RND) or *nanomaterials by design*, was discussed by many experts (Burello &Worth, 2011; Nicholls et al, 2011). One possible multi-faceted approach is illustrated in **Figure 2**, reflecting an engineering, software-lead, market-oriented mind-set (Mize, 2004).

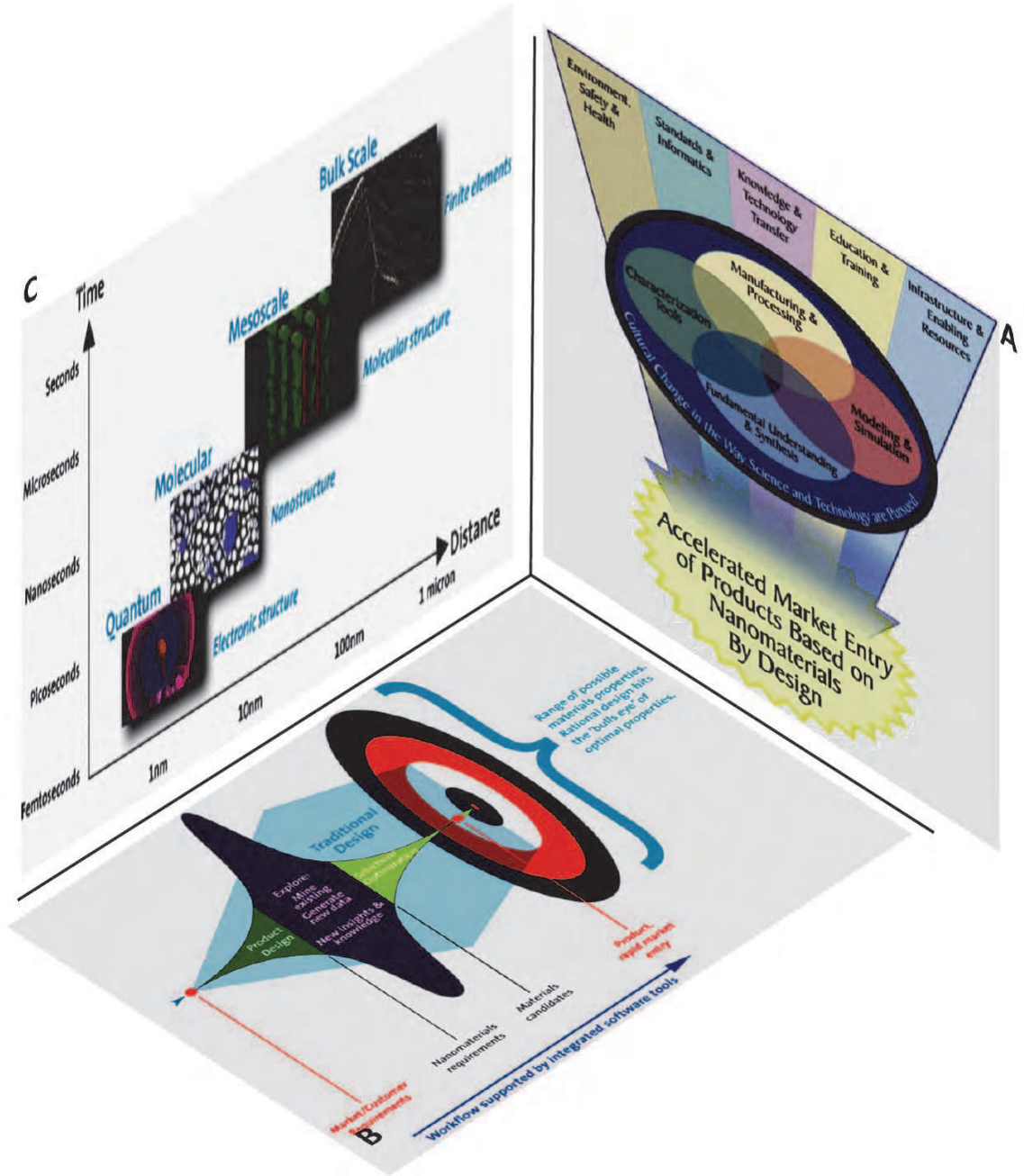


Fig. 2. The rational nanomaterials design (RND) complex process integrates the modeling-simulation methods with the theory, experiment, and converts resulting data into knowledge, then used in nanofabrication (from Mize, 2004).

The concept of *nanomaterials by design*, or *nanomaterials rational design* (NRD) refers to the ability to employ scientific principles in deliberately creating structures with nanoscale features (e.g., size, architecture) that deliver unique functionality and utility for target applications. While advances in technologies including synthesis, manufacturing, and characterization are very important factors in realizing this vision, an overarching strategy

and rational design framework is essential. This involves the integration of modeling and simulation methods with theory, experiment, and the transformation of the resulting information into knowledge, which is then applied in processing and manufacturing. Key to this approach is the use of dedicated software — modeling, simulation and informatics (Barnard, 2010; Schommers, 2007).

In Figure 2, each plane-panel is one facet of NRD concept. Fist, the NRD algorithm of *accelerated product market entry* is charted in panel **A**. Next, the traditional manufacturing process is compared with the NRD in panel **B**, using the metaphor of *hitting the bull's eye of a target*. Finally, several modeling and simulation methods in panel **C** are addressing a range of sizes, suited for hierarchical materials (edited from Mize, 2004).

Thus, the panel **A** outlines a "cultural change in the way science and technology are pursued", a system leading to "accelerated market entry of products based on nanomaterials by design ".The goal is creating a nano-material based product able to deliver unique functionality and utility, for some very specific, pre-designed *targeted* applications.

Next, the panel **B** illustrates, in same figure, the process of Rational Nanomaterials Design. Herein, a *traditional* product manufacturing process is compared with a *rational* approach, using the metaphor of preparing to hit a *target*, in this case the *product rapid market entry.* Here, the traditional process, shown in light blue, results in more of a "hit and miss" approach, with more products missing the exact target than compared to the rational approach.. By contrast, the rational approach goes straight to the center of the target, hitting the "bull's eye" of optimal properties (Mize, 2004).

Finally, the panel **C** from figure 2 above, charts the different modeling and simulation methods to address the range of sizes of a complex material, from nano up to micro dimensions. The modeling of molecular systems was lately enabled by the unprecedented capabilities of super computers. This high performance in turn has driven important algorithmic advances, to a point where advanced calculations can be carried out from our desktop computers (Mize, 2004).

Molecular modeling and simulation combines methods that cover a range of size scales (the sub-atomic quantum mechanics; the atomistic level of molecular mechanics methods, the micrometer-scale mesoscale modeling) in order to study material systems. It is extremely expensive in terms of computing power to apply the more fundamental methods. Each step up the length scale offers the ability to model larger and more complex systems, with the tradeoff of a greater level of approximation in property prediction (Nicholls et al, 2011; Thamwattana et al, 2010; Vasiliev et al, 2009)

The modeling and simulation efforts are still exploding in different areas, often far apart, thus somewhat difficult to process, especially by a first-time reader. Thus, for their benefit, we decided to focus our discussion on modeling and simulation, to one major hub, namely the NSF-funded Network for Computational Nanotechnology (NCN), including its web-based interactive educational portal (www.nanoHUB.org).

2.2 Modeling and simulation

Molecular modeling is the materials representation at atomic- molecular level with 3D computer graphics, *via* graphical mathematical descriptions of the system. This allows scientists to predict fundamental relationships between structure, properties, behavior, composition, and the external stimuli. *Simulation* is the use of a computer to apply these methods to imitate the behavior of a real system, leading to a better understanding of that

system. It can allow the prediction of properties of complex systems with many different discrete parts (Barnard, 2010; Batelle Institute, 2007; Mize, 2004).

2.3 Case study: The three dimensional nanoelectronic modeling

Device physics and material science meet at the atomic scale of novel nanostructured semiconductors, and the distinction between a new ultra-small material and a device is frequently blurred. The quantum–mechanical effects in the electronic states of the device and also the granular atomistic representation of the underlying material are equally important. In some instances, the approaches based on a continuum representation of the underlying material typically used by device engineers and physicists become invalid. *Ab initio* methods currently used by material scientists typically do not represent the band gaps and masses precisely enough for device design, or they do not scale to realistically large device sizes. The plethora of geometry, material, and doping configurations in semiconductor nano-devices suggest that *a general nano-electronic modeling tool* would be highly desirable (e.g., Qiao et al, 2006; Klimeck et al 2007a; 2007b).

The 3-D NanoElectronic MOdeling (NEMO 3-D) tool was pioneered by Klimeck and others, to address these needs and was recently expanded to NEMO 5 (**Figure 3**). Based on atomistic valence force field and a variety of nearest neighbor tight-binding models (e.g., *s*, sp^3s^*, and $sp^3d^5s^*$), NEMO 3-D enables the computation of strain and electronic structure for about 64 and 52 million atoms, corresponding to $(110\ nm)^3$ and $(101\ nm)^3$ volumes, respectively (Klimeck et al, 2007a, b).

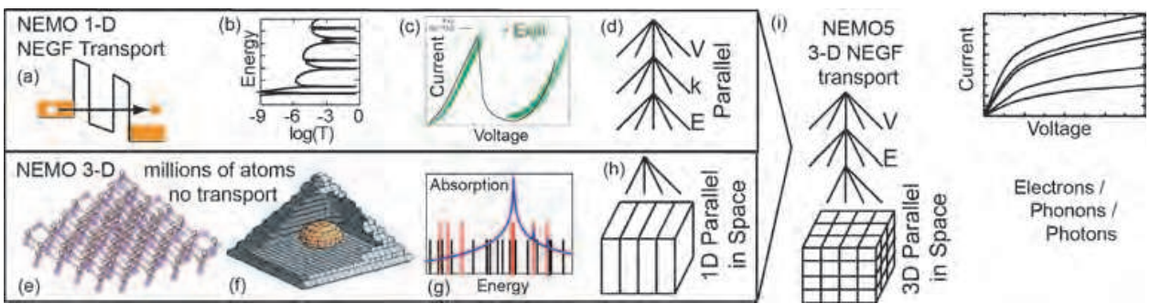


Fig. 3. The NE*M*O5 3-D simulation charts the electronic properties of individual atoms into realistic structures with millions of atoms, and computes relevant nanostructure properties such as strain relaxation, phonon modes, electronic structure, quantum transport etc (reproduced from Klimeck, 2011a by kind author's permission). More details in text.

The physical problem may involve very large scale computations and NEMO 3-D has been optimized to be scalable from single central processing units to large numbers of processors on supercomputers and clusters. NEMO 5 is a parallel multiscale tool for nanoelectronics, allowing among others, simulation of quantum transport in nanodevices using the non-equilibrium Greens function (NEGF) and open-boundary wave-function formalisms. NEMO 3-D and NEMO5 have been released with an open-source license (in 2003, respectively 2011) and are developed by the NCN interactive educational portal (www.nanoHUB.org). Two interesting examples of theoretical models are briefly discussed below, indicative of essential algorithmic and computational components that have been used in the development and successful deployment of NEMO 3-D.

2.3.1 Example: Quantum dots

After much effort in surface chemistry development and optimization by several groups, fluorescent semiconductor nanocrystals probes, also known as quantum dots or Qdots, are now entering the realm of biological applications with much to offer to biologists. The road to success has been paved with hurdles but from these efforts has stemmed a multitude of original surface chemistries that scientists in the biological fields can draw from for their specific biological applications. The ability to easily modulate the chemical nature of Qdot surfaces by employing one or more of the recently developed Qdot coatings, together with their exceptional photophysics have been key elements for Qdots to acquire a status of revolutionary fluorescent bio-probes. Indeed, the unique properties of Qdots not only give biologists the opportunity to explore advanced imaging techniques such as single molecule or lifetime imaging but also to revisit traditional fluorescence imaging methodologies, or based on shifting the diffraction peak, and extract yet unobserved or inaccessible information in vitro or in vivo (Alivisatos, 2004; Asher et al 2002; Cameron, Zhong & Knoll; Cheng et al. 2008; Feng et al, 2007, 2008; Medintz, Mattoussi & Clapp, 2008; Tomczak, et al., 2009).

The improved Qdots semiconductor nanocrystals display longer quenching time compared to conventional fluorescence dyes and size-tunable optical properties, which recommend them for optoelectronic, photonic or bio-labeling uses. The Qdot synthesis typically happens in liquid-phase, the resulting nanomaterial quality being influenced by experimental conditions. Current trends include (i) lowering toxicity for *in vivo* applications by using core-shell architectures (starting with Hines et al, 1996) and (ii) switching to "one pot" green chemistry (Gu, et al, 2004 ; Mekis, et al, 2003). Control over optical properties *via* the size of Qdots is exploited in polymer–Qdots hybrid materials, where designer architectures can be envisioned to match specific applications (Tomczak et al, 2009).

The several still-frames from **Figure 4a** illustrate the simulation and analysis of a pyramid-shaped Qdot using the "Quantum Dot Lab" application. Therein, several powerful analytic features of this tool are demonstrated, including: the visualization of specific 3D wave functions corresponding to discrete energy levels within the quantum dot; rotating the 3D volume of the quantum dot with wave function; scanning through the energy levels of the states inside the Qdot; comparing the absorption curves for different dot sizes (Klimeck & Haley, 2009).

2.3.2 Example: Electron density in a circular silicon nanowire transistor

Continued down-scaling of transistors have enabled the tremendous advances in consumer electronics. We are reaching the limits where the individual transistors or on-off electron valves are only a few nanometers in diameters wide. At these atomic length scales the electrons do no longer act like billiard balls but like waves. Sophisticated modeling engines that consider a quantum mechanical description of the electrons, an atomistic description of the material, and non-equilibrium electron distributions are needed for device design and optimization.

The "Nanowire" tool on nanoHUB.org enables modeling and simulation of such device, as illustrated in figure **4b** and enables the visualization of the electron density in such ultra-scaled structures through 3D volume rendering. In each panel, the left and the right regions on the image represent the large electron densities in the source and drain of the transistor. The central section represents the gated region that enables the control of the electron flow through the nano-scale on-off switch (Klimeck & Mehrotra, 2011; Park et al, 2011).

In the following sub-chapter, we will examine the **organic-inorganic hybrid nanomaterials**, specifically conductive polymer hybrids for enhanced sensing and actuation.

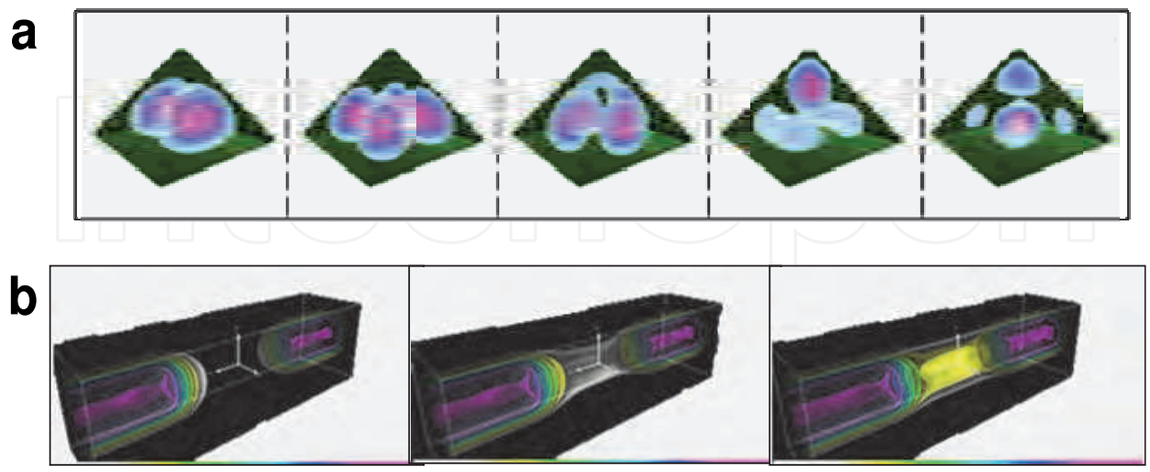


Fig. 4. Examples of using the NEMO 3-D tool. (**a**)Video frames of the simulation and analysis for a pyramid-shaped Qdot using Quantum Dot Lab; (**b**) Electron density in Si nanowire transistor (from Klimeck & Haley, 2009; Klimeck & Mehrotra 2011 reproduced with kind permission from the author). See text for more details.

3. Hybrid organic-inorganic material interfaces

Next, we will discuss aspects of organic-inorganic hybrid materials an important class of *functional* nanomaterials. Many of which are designed to be sensitive to the environment, so-called *responsive* or even *intelligent*. Interestingly, these nano-matrices bring a rich blend of complementary attributes, e.g., optical, photocatalytic, electrochemical, mechanical properties, that are tailored for specific applications in nanoelectronics, plasmonics, bio/catalysis, fuel cells, diagnostic imaging, etc**.** (Dong et al, 2005, 2006, 2009; Feng et al, 2008; Kickelbick, 2007; Gomez-Romero & Sanchez, 2004 ; Ruiz-Hitzky, Ariga & Lvov, 2007; Sanchez, et al, 2011; Vivero-Escoto & Huang, 2011; Willner, Willner & Katz, 2007).

3.1 Inorganic and organic molecular components

Hybrid (bio)organic–inorganic materials offer opportunities for both basic research and new exciting applications, *via* their inherent multifunctional properties. Today's design, tailoring of complex hybrid systems, is possible *via* cross-disciplinary, synergistically coupled approaches in biomolecular engineering, smart processing or nanofabrication.

The impressive set of inorganic-organic hybrid nanomaterials span a wide spectrum of properties, yielding innovative applications in areas such as plasmonics, nanoelectronics, health, energy, the environment among others (e.g., Ruiz-Hitzky, Ariga & Lvov, 2007; Knoll et al, 2004; Li et al, 2007; Nakamura, Katagiri & Koumoto, 2010; Peng et al, 2007, 2008; Stemmler et al, 2009; Vivero-Escoto & Huang, 2011).

Today the main hybrid materials that find applications in industry are based mostly on the association between metal oxides or metal–oxo polymers and organic molecules or macro-monomers of all kinds including bio-components. An exhaustive description of all the

chemical reactions involved in the construction of organic and inorganic components is beyond of the scope of this work and the reader is referred to several reviews and books (Boissiere, et al., 2007; Cong & Yu, 2009 ; Dong, et al., 2008; Kickelbick, 2007; Romero & Sanchez, 2004 ; Mullen, et al., 2008; Ray, et al., 2005). A brief outline on the synthetic nanofabrication of the *inorganic* and the *organic* components follows.

3.1.1 The inorganic component

Typical inorganic partners in the hybrid include noble metal nano-objects (Coffinier et al, 2010; Ghodbane et al, 2010), magnetite (Fe_3O_4) nanoparticles, solid or mesoporous silica or quantum dots. The inorganic metal–oxo polymers or metal oxides (Hongqin et al, 2009) are typically produced as an amorphous or nanocrystalline network, or metal–oxo cluster *via* condensation of metal organic precursors or salts. The sol–gel polymerization can be driven through hydrolysis reactions (addition of water to reactive precursors such as alkoxides, or chemical or thermal modification of the pH of aqueous solutions containing metallic salts) to form reactive M–OH species that condense yielding metal–oxo oligomers and polymers assembled via M–O–M and/or M–OH–M bridges. Metal–oxo species can also be generated through thermal elimination of organic moieties. The elimination of ester, ether, and/or alkyl chloride are well known examples of using "sacrificial" routes to produce metal-oxides through thermally induced non hydrolytic sol–gel chemistry (Escribano, et al., 2008 ; Ohara, 2011 ; Pyun, et al., 2001 ; Shen & Shi, 2010 ; ten Elshof, et al., 2010 ; Yamada, 2009; Yao, Gao & Yu, 2010; Yuan & Muller. 2010).

3.1.2 The organic component

The organic part is usually a polymer, carbon nanotube, a biomolecule, etc. Organic components can be introduced into an inorganic network in two different ways, as network modifiers (molecules) or network formers (macromolecules). The most commonly used network modifiers or network formers are coupled to inorganic moieties through organo silicon alkoxides or chlorides. The introduction of organic network formers into an inorganic network to form hybrid materials can be performed *via* two main strategies. One method is by using already pre-synthe-sized functional macromonomers that are compatibilized with the inorganic component either via chemical grafting "class II" or through embedding with a growing inorganic network in a common solvent to form "class I" hybrid materials; The second approach is *in situ* generation through photo- or thermo- induced polyadditions in presence of a radical initiator, atomic transfer radical polymerization, chemically or electrochemically driven oxidative polymerisation (polypyrrole, polyaniline, polythiophene etc), or polycondensation (polyesters, polyimine, polyamides formophenolic) (Fabregat-Santiago et al, 2011 ; Hu & Shea, 2011; Ma, et al, 2007; Ohara, 2011 ; Vivero-Escoto & Huang, 2011; Weickert, et al., 2011).

3.2 Nanofabrication of organic-inorganic hybrid nanomaterials

In the same line of environmentally friendly, mild synthetic methods for hybrid nanocomposites, a hot research topic in material science is the development of bio-inspired hybrids. Synthetic strategies for functionalized inorganic-organic hybrids based on self-assembly strategies, whether in-situ, template-induced, evaporation-induced, layer-by-layer, etc were recently reviewed (Cong et al, 2009). The "soft chemistry" (*la chimie douce*) approach to synthesize various organic-inorganic nanomaterials was examined (Sanchez et al, 2011)

from sol–gel-derived and nano-building blocks-based hybrids, to thin films of nano-structured porous materials and aerosols. The importance of characterizing the hybrid interface was highlighted, by using multiple modern techniques such as diffusion-ordered spectroscopy nuclear magnetic resonance or ellipsometry.

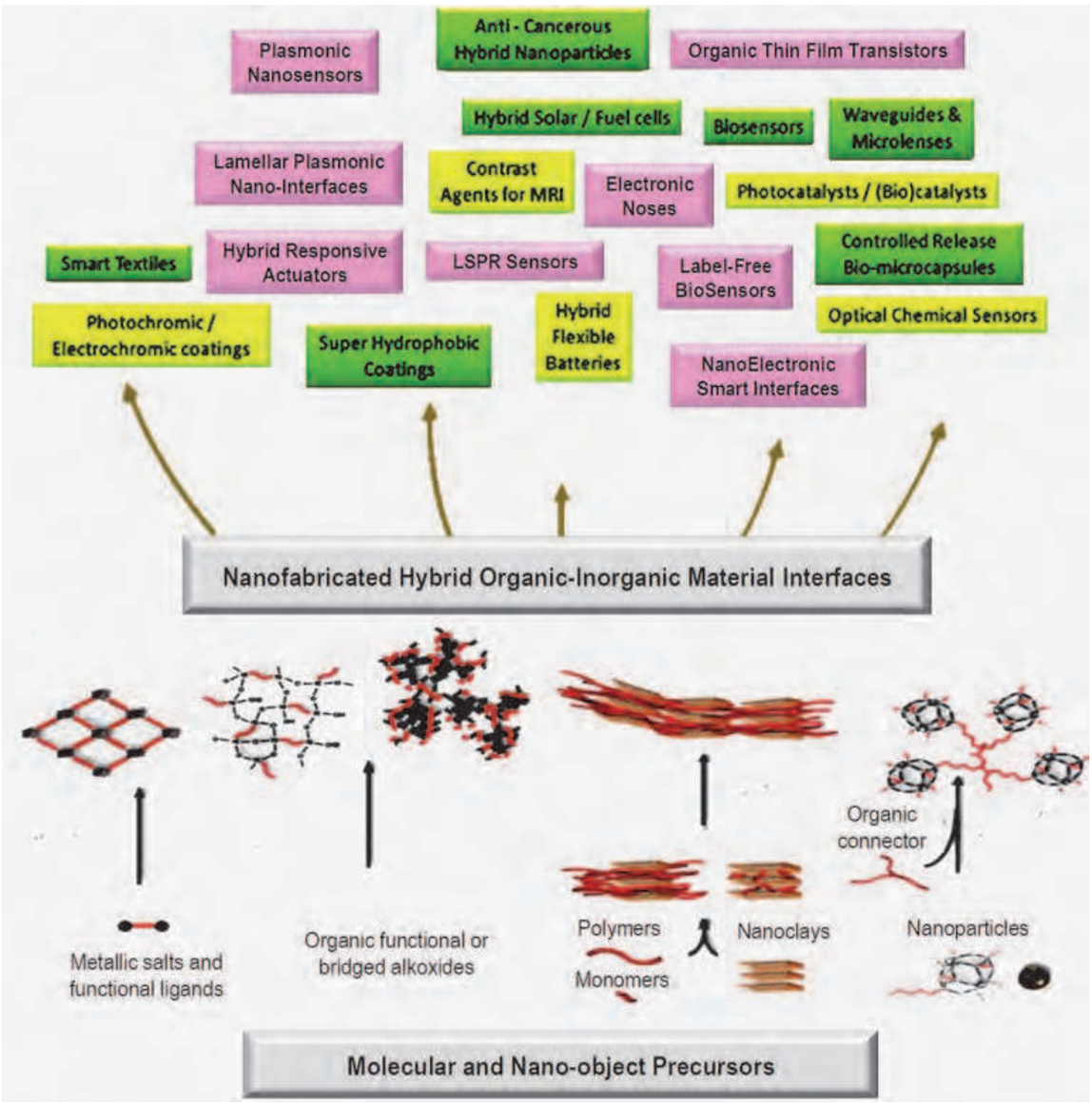


Fig. 5. The process of micro- and nano-fabricating of real life applications starts at the bottom, with the synthesis and self-assembly of molecular and nano- precursors into smart hybrids. Subsequently, these nanofabricated, hierarchically structured, hybrid bio/materials are employed to manufacture real devices by using of complex interactions, primarily from physics, chemistry and bioprocessing (reproduced in part from Sanchez, 2011 by kind permission of the Royal Society of Chemistry).

3.3 Hybrids based on nanoscale conductive polymers for enhanced sensing and actuation

The synergistic integration of similar-size biomolecules and nanomaterials resulted recently in novel cross-bred bio-nano-materials displaying an exquisitely unusual set of electronic, photonic, catalytic and recognitive properties and functionalities. More specifically, the electroactive nano-scale polymers have been integrated with receptors, enzymes, antibodies, whole cells, and or nucleic acids in their matrix, leading to uniquely advanced biosensors and bioelectronics.

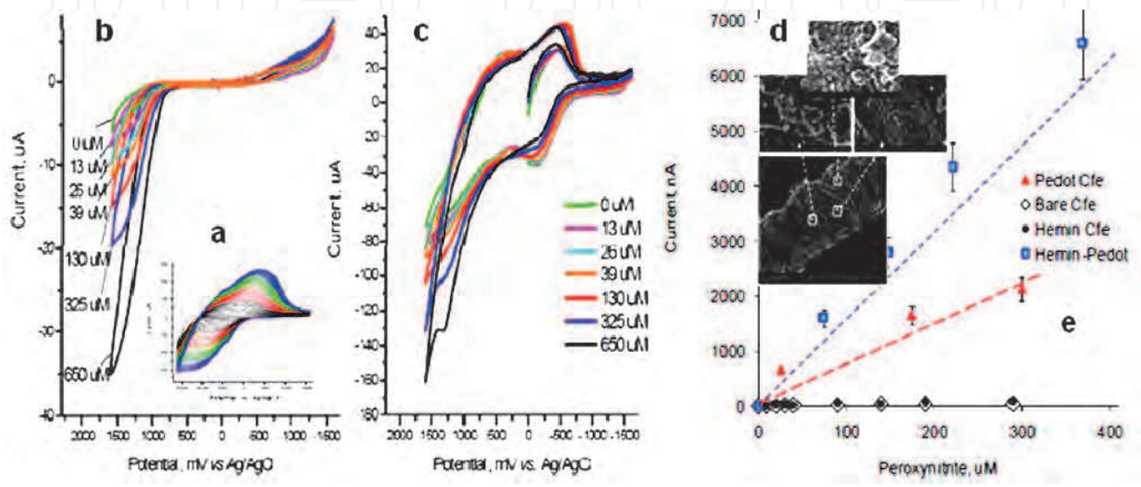


Fig. 6. The PEDOT–nanostructured interface significantly enhanced electrochemical sensing of a reactive nitrogen species as analyte. The panels represent: (**a).** inset showing the catalytic interface electro-deposition from solution; (**b).** Cyclic voltammogram for hemin-modified; (**c).** Same conditions, for hemin-PEDOT-modifed, after addition of different aliquots; (**d).** PEDOT nanostructure as evidenced by SEMs; **€.** Increased biosensor sensitivity after adding PEDOT to the catalytic interface (reproduced from Peteu et al, 2010 by kind permission of Elsevier).

Moreover, the advanced use of conducting polymers in sensors and actuators has grown over the past decade, also due to their compliance with nano-fabrication. As a result, their reversible, strong biomolecular interactions at nanoscale translated into (i) biomolecular sensing with lower detection limits and enhanced sensitivity and also into (ii) nano-actuating materials (Kulesza, et al, 2006; Lee, et al., 2008).

Thus, electroactive polymers based on polypyrrole or polyethylene dioxythiophene (PEDOT) were employed to prepare electrochemical or optochemical bioanalytical sensors, including at nano scale. These 'synthetic metals' exhibit high conductivity, mediate fast transfer of charge carriers and can be synthesized under mild conditions, through simple deposition onto conductive surfaces from monomer solutions with precise electrochemical control (Liao, Huang &Li. 2009 ; Muller, et al., 2007 ; Rahman, et al., 2008).

More specifically, by comparison with polypyrrole, PEDOT seems to show a better electrochemical stability, with a better conservation of its conductivity and charge. Also, PEDOT has a higher ionization potential, protecting better against the oxidative damage. These soft, synthetic metals were proven to enhance nano-sensing or deliver nano-actuation. Recently, one group reported a 50-100 times increase in sensitivity for peroxynitrite

detection, as a result of adding the nano-structured polyethylenedioxythiophene (PEDOT) to a catalytic electrochemical detection matrix (Peteu, et al, 2010), as outlined in **Figure 6**.

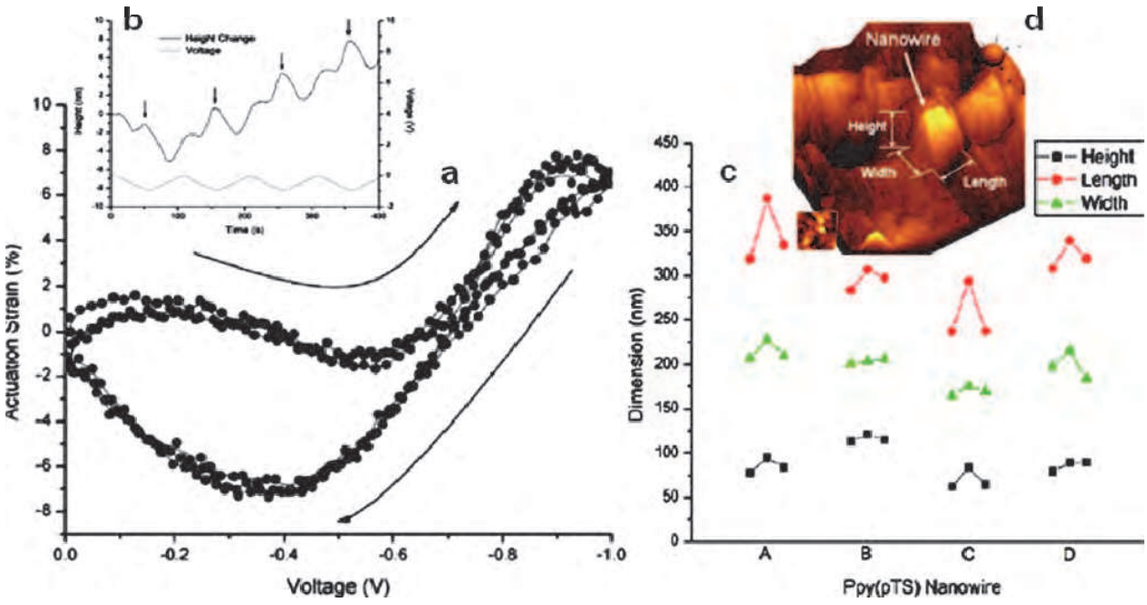


Fig. 7. The actuation of the 50 nm diameter polypyrrole nanowires. **(a)** Real-time actuation analysis for the 50 nm diameter polypyrrole-polytoluenesulphonate nanowire as the voltage is cycled 0 to −1 V. As expected, the nanowire shows cyclical increase and decrease behavior, with the greatest change in height at −1 V; (**b)** Actuation strain plot for 50 nm diameter PPy(pTS) nanowire confined in a polycarbonate membrane as the voltage is cycled from 0 to −1 V; (**c**) Three-dimensional AFM rendering showing a PPy(pTS) nanowire protruding from the surface of the polycarbonate membrane, its surface chemically etched to expose more of the embeded nanowires; (**d)** Analysis of four, 50 nm diameter, PPy(pTS) nanowires. The three points in each data set correspond to the initial oxidizedstate, reduction, then reoxidation of the nanowires (reproduced from Lee et al, 2008, with kind permission by Institute of Physics).

The inherently conductive nanoscale polymers molecules can designer-hybridize with inorganic nano-components leading to tailored, bottom-up nanofabricated, materials with unique properties. Amongst these are nano-scale or nano-structured actuators allowing the conversion of chemical, electrical or thermal energy/ sources into mechanical energy. These so called artificial muscles have found nano-scale applications including for nanorobots or responsive release of countermeasures (Liu, et al, 2010; Tamagawa, et al, 2011).

Additionally, nanoscale actuators are expected to become a major area of development within nanofabrication. They are essential components of the NEMS and nanorobots of the future, and are poised to become a major area of development within nanoscience and nanotechnology. As illustrated in **Figure 7**, our group has reported for the first time the actuation of individual 50 nm diameter polypyrrole nanowires, when triggered electrochemically in solution, by volume change as the result of its oxidation state (Lee et al, 2008).

In the following sub-chapter, we will examine the **plasmonics methods and structures**, with focus on lamellar plasmonic nano-interfaces for optical sensors.

4. Nanoplasmonic interfaces

The last decade was marked by exciting discoveries and advances in two young scientific fields that concern optically active nanostructures: *nanophotonics* and *plasmonics*. Nanophotonics represents the study of interactions between light and nanostructured matter, which occur at subwavelength ranges and are determined by the very specific physical, chemical and structural properties of nano- matter. The spotlight herein will be on plasmonics and its applications. However there are several reviews and books available with focus on nanophotonics for those interested. (Eustis, S. & El-Sayed, 2006; Stewart et al, 2008; Willets & Van Duyne, 2007)

Current research in *nanoplasmonics* is directed towards understading through the effects of nanostructures on surface plasmon resonance (SPR), finding new nanomaterials, such as periodically nanostructured thin films or metamaterials and nanoporous silicon layers for increased sensitivity and for supporting excitation using THz frequency range rather than the visible wavelength band. Future research will continue to be directed also towards hyphenation of plasmonic sensors with other techniques such as interferometry for better sensitivity or for obtaining complementary information (Kim, et al., 2008; Sannomiya & Voros, 2011).

4.1 Plasmonic metamaterials

The rapid development of synthetic nanofabrication for complex, nanoscale metal structures has led to the emergence of the field of plasmonics, exploring the local and far fields around small metal particles. Additionally, it is concerned with the use of particle morphology as a means to control and tune these fields and with the way energy is dissipated and transported through these structures. It is centered on optical properties of metallic nanostructures and their use for manipulating light at the nanoscale (Ahl et al, 2008; Galopin et al, 2009; Gitsas et al, 2010; Grosserueschkamp et al, 2009; Zhou et al, 2010) and some plasmonic nanomaterials are illutrated in **Figure 8**.

Plasmonic metamaterials, exhibiting simultaneously negative dielectric permittivity and magnetic permeability in a given frequency range, hold the promise for significantly increasing the field depth in imaging applications as well as for a higher sensitivity to changes in the bulk solution. (Mayer & Hafner, 2011; Stewart et al, 2008).

4.2 Nanoplasmonic sensitive interfaces

Another interesting recent achievement concerns the nanofabrication of a coherent nano-metallic light source, or surface plasmon amplification by stimulated emission of radiation, or SPASER, consisting in a 44-nm diameter nanoparticle with a Au core surrounded by a shell of dyed silica which acts as a gain medium possible applications including magnetic data-storage industry, nano- lithography, probing and microscopy (Bergman & Stockman, 2003; Noginov, et al., 2009). Some very exciting highlights of nanoplasmonic sensitive interfaces include the plasmonic nanoholes, the surface-enhanced Raman spectroscopy (SERS), the localized surface plasmon resonance, or the localised surface plasmon fluorescence (Galopin et al, 2009a; Tripp, Dluhy & Zhao, 2008). These and others are employed for detection or imaging and will be briefly discusses below.

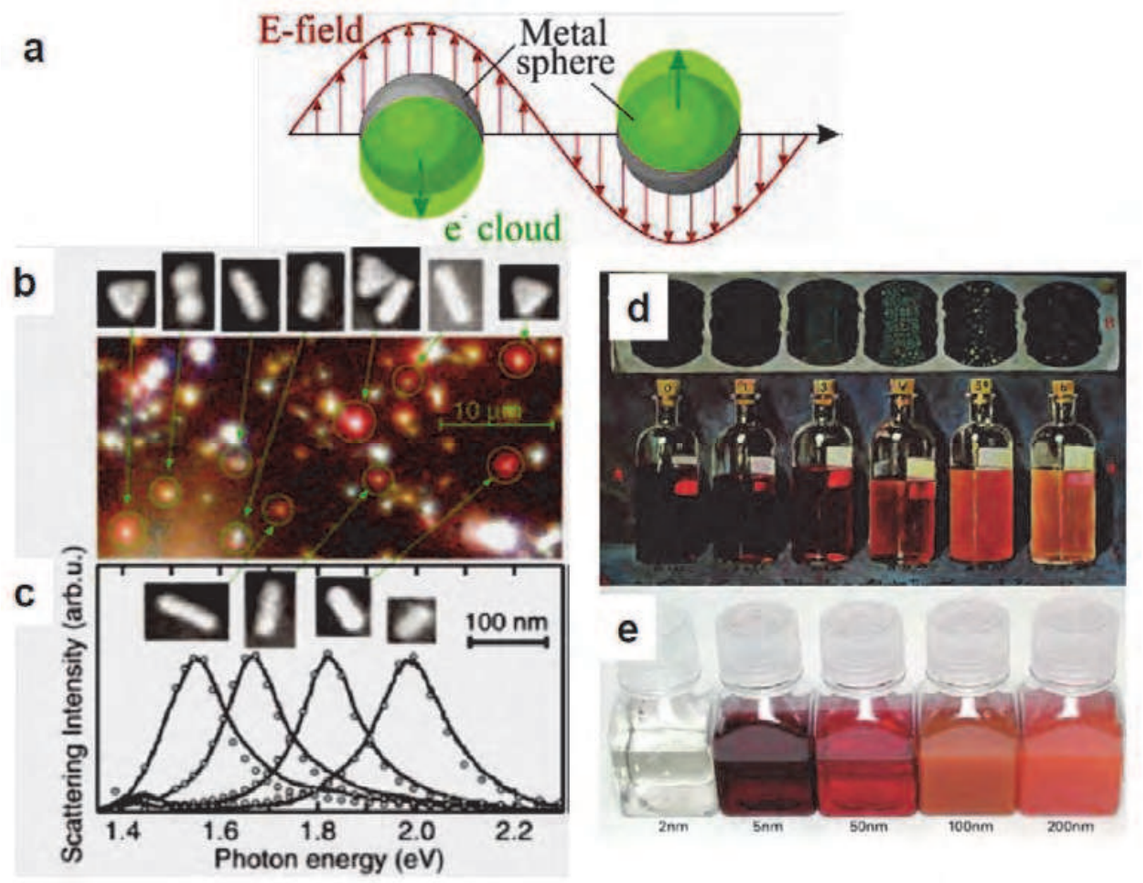


Fig. 8. Plasmonics nanomaterials. (**a**) Localized surface plasmon of a metal sphere showing the displacement of the electron charge cloud relative to the nuclei. (**b**) Dark-field microscopy image and corresponding SEM images; (**c**) light scattering spectra of Au nanocrystals of different shapes; (**d**) illustration of Dark field scattering of Au colloids of various sizes from year 1909); (**e**) actual photo of similar colloid solutions (reprinted from Kelly et al, 2003 with kind permission from American Chemical Society, from Kuwata, et al., 2003 with kind permission American Institute of Physics and from Mayer & Hafner, 2011 with kind permission from the American Chemical Society).

The discovery in 1998 of extraordinary optical transmission (EOT) in arrays of subwavelength nanoholes in a metallic film (Ebbesen, et al, 1998) led to important progress in instrumental design for SPR biosensing, and improvement of sensitivity of both detection and imaging (Lindquist, et al, 2009). Designs based on nanohole arrays served to monitor the binding of organic and biological molecules to the metallic surface or to the spectacular observation, with the naked eye of protein monolayer formation. In an interesting experimental setup, nanoholes served a dual purpose: as the optical active element for analyte detection and as nanochannels facilitating analyte mass transport to the active surface (Eftekhari et al , 2009).

New exciting possibilities for biodetection using plasmonic devices arise from the localized surface plasmon resonance (LSPR) phenomenon displayed by nanomaterials (such as metal nanoparticles, nanoshells,nanodisks, nanowires etc (Lu, et al, 2009; Praig et al, 2009; Zhou et

al, 2008) as a result of interaction of light with particles much smaller than the incident wavelength. A result derived from LSPR studies is the advance in highly sensitive surface characterisation methods such as SERS, in particular very sensitive, and producing a specific signal, or fingerprint of certain molecules. For example, the *in situ* cell pH sensor was mapped *in situ* with SERS and gold nanoparticles functionalized with 4-mercapto-benzoic acid. One consequence of LSPR is the significantly higher resulting electric field, which offer opportunities to improve the sensitivity of the detection (McFarland, et al, 2003; Raschke, et al. 2003; Baciu, et al., 2008)

Although LSPR has a short penetration depth of the electric field (around 20 nm), the intensity of SPR signal is strongly dependent on the shape, size, material nature and architecture of nanoplasmonic devices, active research being currently focused on understanding and controlling this relationship. LSPR biosensors include label-free detection of the biomolecular interactions, such as antigen–antibody reactions, DNA–DNA or PNA–DNA hybridizations to monitor cell activity by measuring cell metabolites and sensitive detection small analytes (e.g glucose) or antibodies Besides LSPR, localized surface plasmon coupled fluorescence was also exploited to obtain highly sensitive biosensors (Endo, et al., 2006, 2008; Yamamichi, et al., 2011; Zhou et al, 2009).

4.3 Lamellar plasmonic nanointerfaces for optical sensing

Plasmonics applications include high performance near-field optical microscopy (NSOM), high resolution imaging, targeted drug delivery, biosensors, catalysis, solar cells. Also, it is a preferred approach to attain exquisite precision in controlling optical processes. Recent investigations encompass a wide range of examples of interactions between light and nanostructured matter, from guiding the light through metal nanowires below the diffraction limit to optical lenses formed by a thin film of metal, components of metamaterials (artificial materials from nanoscale building blocks), or lamellar plasmonic nanointerfaces for optical sensors (e.g., Galopin, et al., 2010 ; Niedziolka-Jonsson, et al, 2010; Szunerits, et al., 2008a; 2008b; 2010; Touahir et al., 2011)

In particular, the nanostructured noble metals exhibit an intense optical near field due to surface plasmon resonance (SPR), therefore promising widespread applications and being of interest to a broad spectrum of scientists, ranging from physicists, chemists, and materials scientists to biologists. A versatile, highly-sensitive detection of DNA hybridization is described using metal nanostructures-enhanced fluorescence (MEF) emission intensity when fluorescently-labeled DNA oligomers are covalently immobilized on a nanometer-thin amorphous silicon–carbon layer capping the metal nanostructures (Touahir, et al, 2010).

The MEF structures are formed by thermal deposition of silver, gold or silver/gold thin films on glass surfaces and post-annealing at 500 °C. The choice of the metal film allows for tuning the optical properties of the interface. The metallic nanostructures are subsequently coated with an amorphous thin silicon–carbon alloy (a-Si0.80C0.20: H) layer deposited by PECVD. Carboxydecyl groups are attached on these surfaces through hydrosilylation then reacted with amine-terminated single-stranded DNA oligomers, forming a covalent link. The immobilized DNA is hybridized with its complementary strand carrying a fluorescent label. Through optimization of the thickness of the a-Si0.80C0.20: H alloy overlayer and by working close to resonance conditions for plasmon and fluorophore excitation, the hybridization of very dilute oligomers (5 fM) is easily detected, and the hybridization kinetics can be monitored in situ and in real-time as illustrated in **Figure 9**.

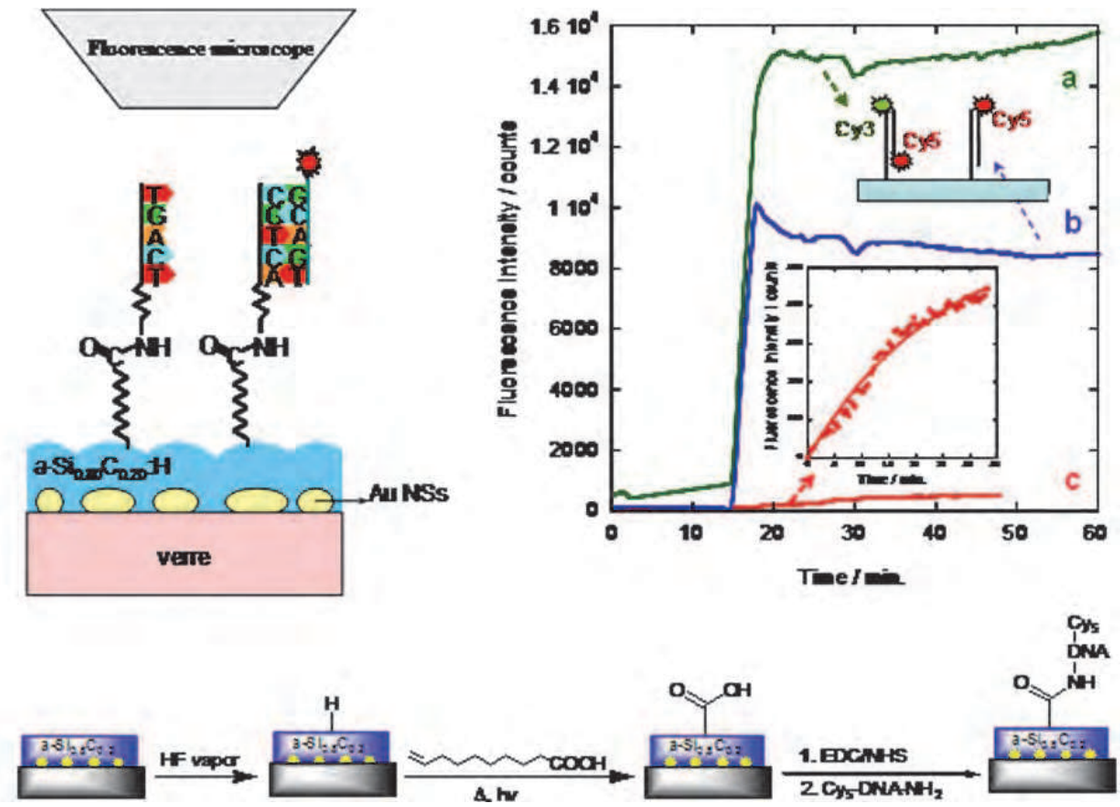


Fig. 9. Lamellar plasmonic sensor (**A**) Schematic of the LSPR-metal enhanced fluorescence structure and set up (**B**) Surface functionalizing reaction scheme. (**C**) Fluorescence intensity of Cy5-ON targets hybridized with immobilized probes (**a**) and probes (**b**). Curve (**c**), enlarged in the inset, correspond to the hybridization of Cy5-ON' targets with immobilized probes. The sensor is obtained from structure 4 coated with 5 nm *a*-$Si_{0.8}C_{0.2}$: H. An exponential fit of curve (c) is shown in the inset (reproduced from Touahir, et al, 2010, with kind permission from Elsevier)**.**

Coating metal nanostructures with a nanometric thin film of amorphous silicon–carbon alloy allows for designing an efficient sensor, which exhibits a good stability in conditions typical of biological assays and with efficient covalent attachment of the biological probes. Such a sensor exhibits a high sensitivity, allowing for the detection of trace amounts of DNA and the investigation of hybridization kinetics in situ and in real-time, in a spatially-resolved, classical geometry of epi-fluorescence.

Such investigations do therefore not require the setting up of custom made, highly accurate instrumentation, which should make this type of measurements easier and help in determining the respective roles of the various factors coming into play for determining hybridization kinetics at solid surfaces. Another attractive characteristic of these substrates is the capability of recording images of labeled fluorescent probes with a high sensitivity, while simultaneously monitoring the interaction of the probes with non-labeled species present in the assay through LSPR imaging. This capability should help in analyzing phenomena like competitive interactions which come into play in realistic diagnostic assays (Barka, et al., 2011**;** Touahir, et al, 2010; Khor et al, 2007)**.**

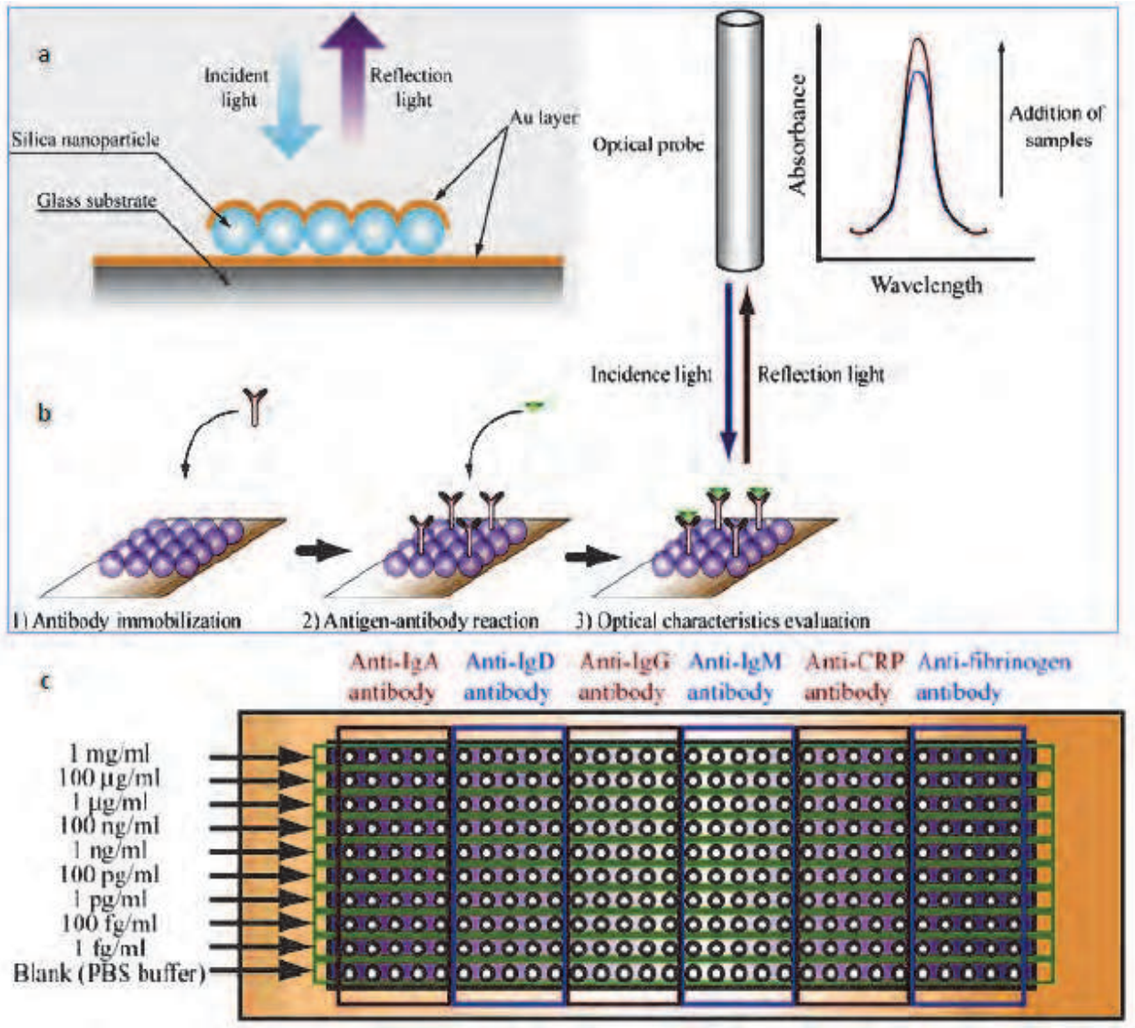


Fig. 10. LSPR-based, core-shell nanoparticle layer device for label-free multi-detection of antigen-antibody reaction. (a) Schematic of the LSPR-based nanochip. The surface modified silica nanoparticles were aligned onto Au deposited glass substrate, with the Au film subsequently deposited onto the silica nanoparticle layer. (b) Experimental setup of the multi-array chip. (c) Chip design illustrating antibody immobilized spots and antigen concentrations. Six kinds of antibodies and antigens were spotted onto the multi=array LSPR-based nanochip surface (reproduced from Endo et al, 2006, with kind permission from the American Chemical Society).

The past decade has witnessed an exciting research effort directed toward the development of hybrid plasmonic interfaces, driven mainly by convincing applications in SPR and LSPR sensors and enabled by rapid progress in nanotechnology and nanoscale science, which in turn facilitates their successful synthesis and characterization.

In the following sub-chapter, we will examine the nanoelectronic interfaces , with focus on organic thin film transistors with molecular scale label free detection.

Before LSPR sensors can become high-throughput laboratory and clinical screening tools to compete with ELISA and other common assays, the technique must be parallelized to

handle large numbers of samples. **Figure 10** illustrates an exciting display of the potential of this technology. An LSPR-based multi-array chip was nano-fabricated by depositing an array of 300 spots of nanoliter volumes of antibody solutions on a film of gold capped nanospheres. A scanning optical probe was employed to measure the optical absorbance of the film, to compare the affinity of each antibody for the analyte. The change in integrated absorbance was measured, rather than an LSPR peak shift. Antigens were detected for concentrations in the region of 100 pg/mL (Endo, et al., 2006).

5. Nanoelectronic Interfaces

5.1 Nanofabrication and materials for nanoelectronics

Nanoelectronic devices are based on structures whose material properties vary on an atomic length scale. Such structures can be created with a variety of experimental methods in a variety of different material systems. Interfaces between different materials can be atomically abrupt. These abrupt interfaces enable device designers to confine electrons quantum mechanically. Interactions/transitions between different man-made quantum mechanical states open a cornucopia of new device applications.

Meanwhile, a model of an organic semiconductor nano-device is illustrated in **Figure 11**. This model from fig. **11a** shows the ultimate miniaturization of nanoscale logic circuits. An electron, entering at the lower left electrode, can be directed with varying degrees of probability, to one of the three output electrodes in the upper part of the picture. An aromatic molecule (naphthalene) bonded to four gold electrodes (green) by sulfur atoms (blue) and polyacetylene wires. An insulator on the surface prevents cross-talk between the electrodes. Parts of the molecule and electrodes are drawn in brighter (darker) colors, to suggest an active (inactive) state during a particular read-out. The X, Y, Z symbols represent three possible logical inputs, which can be operated on in various ways to produce a quasi-digital logical output (Stadler et al, 2003).

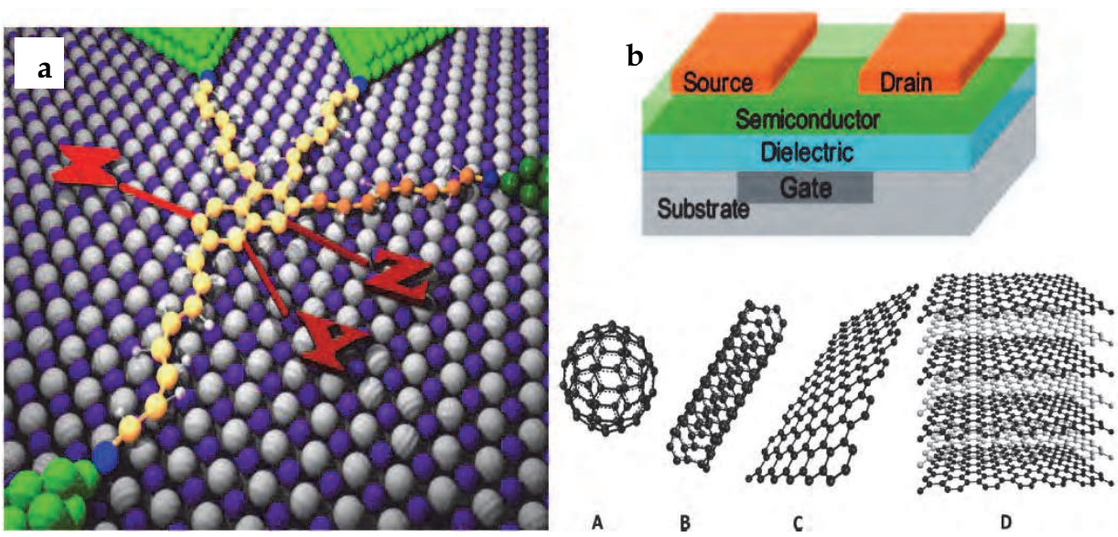


Fig. 11. Organic semiconductor nano-devices. (**a**) Model showing, arguably, the ultimate miniaturization of nanoscale logic circuits (reproduced from Stadler et al, 2003 with kind permission from Institute of Physics); (**b**) A typical organic thin-film transistor (OFET); (**A-D**) The characteristic forms of carbon, and their dimension. See text for more details.

A typical organic thin-film transistor (OFET) is a field-effect transistors (FET) with organic semiconductors in its channel and is made by depositing thin films. The FET has three essential components, source (S), drain (D) and gate (G), as illustrated in Figure **11b**. The gate controls the carrier movement from the source to the drain. Moreover, current OFETs are investigating the use of nano-structured materials such as carbon, to enhance their response. The characteristic forms of carbon, and their dimension as shown in figure 11 are: fullerenes (0D), nanotubes (1D), graphene (2D), graphite (3D).

5.2 Organic thin film transistors as biosensors with molecular scale label free detection

Rapid and highly sensitive PNA-DNA hybridization assays have attracted enormous attention for a wide variety of applications ranging from genotyping to molecular diagnosis (Huang et al, 2001; Ito et al, 2007; Nakamura et al, 2006; Kelly et al, 2003). Conventional optical detection systems based on microarrays and real-time PCR involve expensive detection protocols, typically requiring a fluorescent dye and optical sources/detectors; however, this method has become the standard technique for quantifying the extent of hybridization between surface immobilized probes and fluorophore-labeled DNA targets (Yameen et al, 2010; Yin et al, 2011).

The flowchart of fabrication of an organic thin film transistor (OTFT) is illustrated in **Figure 12**. This particular OTFT has been nanofabricated as follows (Khan et al, 2011a). On either n++ silicon or polyimide substrates with a thin Al film, a 25 nm PVP-HDA dielectric layer was spin-coated, followed thermal evaporation of a 25 nm pentacene active layer and a 35 nm CuPc passivation layer. Source-drain (S-D) electrodes with a width (W) of 1000 µm and length (L) of 50 µm were deposited through a shadow mask. The flow cell was laminated on top of OTFTs for operation in buffer media and biosensing demonstrations.

On a similar device (Khan et al, 20111b), charge discrimination experiments were performed for bovine serum albumin (BSA)/anti BSA immunoassay formation in aqueous buffer solutions at different pHs using OTFT and SPR sensors. The solid arrows indicate an injection of anti BSA and open arrows indicate exchange with pure buffer solution. The OTFT current response (IDS/IDS-baseline) with time upon exposure to anti BSA (500 nM) diluted in buffer solution at pH 7 (black curve) and at pH 5 (blue curve) while operating at a constant bias (VG = -5 V, VDS =-2 V), as sown in figure **12a**. The SPR response, as a ratio between the minimum resonance angle shift (RAS) and the RAS baseline with time, upon exposure to anti BSA (500 nM) diluted in buffer solution at pH 7 (black curve) and at pH 5 (blue curve) using SPR sensing platform, is illustrated in figure **12b**.

Recent advances in chemical detection research, in part benefiting from the overwhelming progress made in organic electronics, have shown great promise for a viable, low-cost alternative to current optical detection systems. The utilization of organic transistor technology in chemical sensors is particularly encouraging. This simple platform allows for the fabrication of low-cost, large-area, and flexible devices with air stability, low-power consumption, biocompatibility, and facile surface modification for the detection of a wide range of analyte species (Knoll, et al., 2011a; Khan et al, 2010; Roberts, et al., 2008).

Many examples exist for the detection of analyte vapors using similar organic thin film transistor (OTFT) platforms, with numerous reports addressing the ability to identify particular analytes either through the use of a fingerprint response or by incorporating selective detection layers on functional OTFTs. Few examples of chemical detection in aqueous systems have been demonstrated. However, these devices were not selective

toward a particular analyte **(**Knoll, et al., 2011b; Khan, et al., 2011; Roberts, et al., 2008, 2009). A selective, *in* situ detection with OTFTs requires a versatile method for the immobilization of various selective molecular probes within proximity to the active transport channel.

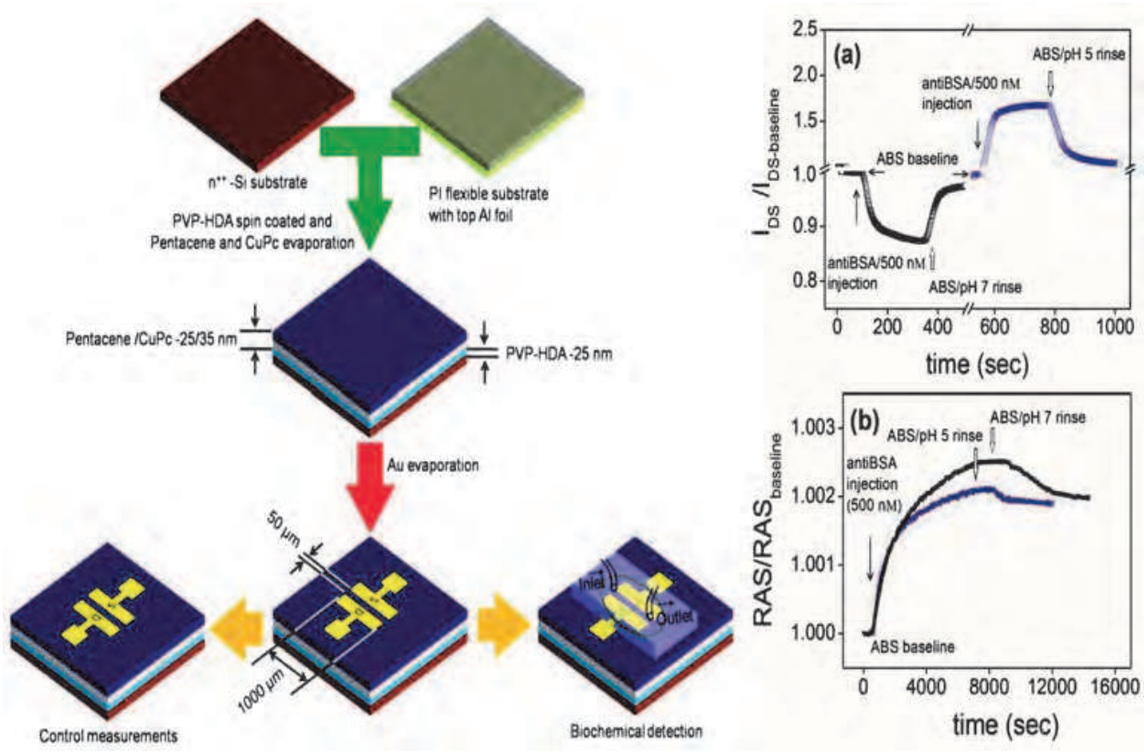


Fig. 12. OTFT sensor fabrication process. Charge discrimination experiments for BSA/antiBSA immunoassay formation in aqueous buffer solutions at different pHs using OTFT and SPR sensors, the solid arrows indicating an injection of antiBSA and open arrows indicate exchange with pure buffer solution, with (**a**) the OTFT current response and (**b**) the minimum resonance angle shift (RAS) response from the SPR sensing platform (reprinted from Khan et al, 2011a and from Khan et al, 2011b with kind permission from the American Chemical Society). See text for more details.

A flowchart of an experimental setup of a carbon nanotube (CNT)-based OTFT sensor and the qualitative, expected detection results are illustrated in **Figure 13**. The use of a target concentration (e.g., 100 nM) of target DNA complement provides excellent discrimination against single/double base mismatches. A Langmuir model can be used to fit the kinetic measurements on the OTFTs sensors based on the PNA-DNA hybridization, which showed that a high affinity constant on the order of $K_A = 4 \times 10^8$ M^{-1} can be achieved in these organic transistor-based detection systems. These OTFT sensors would benefit from the transduction of the DNA binding to an easily read electronic signal.

5.3 Other nanoelectronic devices

Improvement in fabrication will stem from combining bottom-up with "top-down" and self-assembly strategies. New materials such as graphene nanoribbons, organic-inorganic hybrids, inorganic nanowires need to be integrated in

higher-architecture devices. On the other hand, integration of nanostructures in complex systems including micro-fluidics or on Si wafers, etc are extremely important. To this end, the properties at Si-biomolecule interface should be exploited. The nanoelectronics-enabled high computational capacity seems to be critical for future high-throughput screening in clinical medicine or and pharmaceutics. The future nanoelectronic devices should lead to increased functionality and information storage and improve abilities to power and process information from complex in-vivo sensors, drug delivery devices, biomolecules labeling, and protein studies (Yamamoto, et al., 2009).

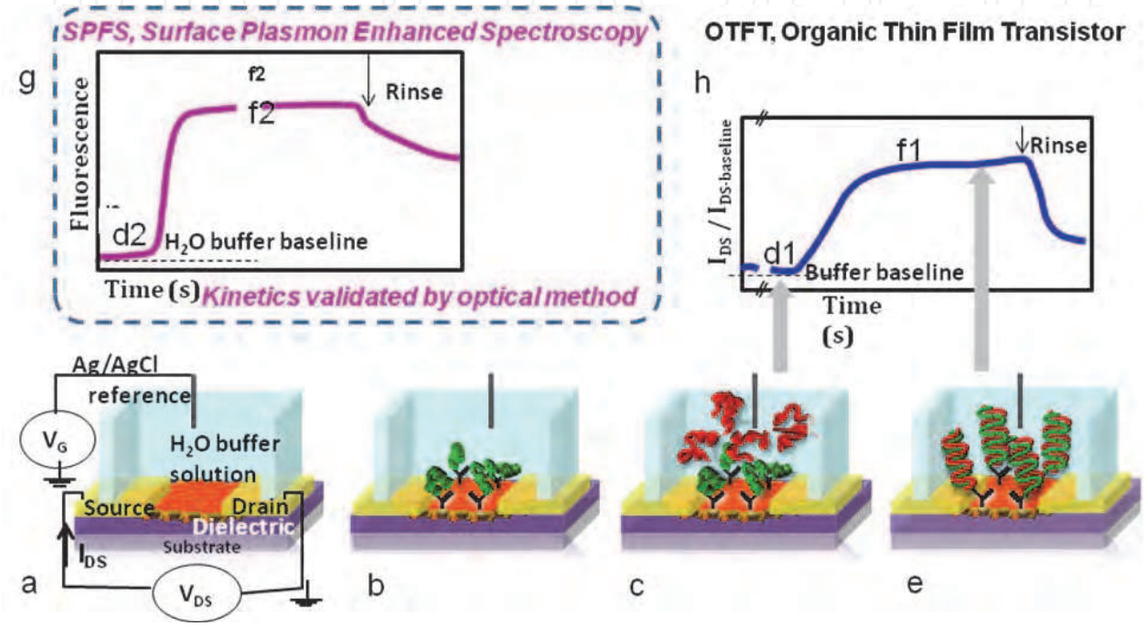


Fig. 13. A model of a carbon nanotube-based, organic thin film transistor sensor. (**a**-**e**) The flowchart schematic of the experimental setup; (**g**-**h**) The expected qualitative detection results for this model.

Several nanoelectronics applications are in the areas of lasers, detectors and/or memory. For *lasers*, the electrons in higher energetic states can emit photons by a transition to lower energetic states. Electrons in *detectors* can be excited by a photon to jump from a lower to a higher energetic state. Furthermore, in the case of *logic memories*, the electrons can transition without loss of energy from one quantum state to the next by quantum mechanical tunneling. The speed of such electronic transport can be extremely high, leading to new low power and high density devices.

The continuing quest for better, faster, inexpensive, smaller devices moved into the nano realm about a decade ago, with spectacular effects, especially visible in the semiconductor industry. This brought along new challenges in *nano-fabrication* including *nano-patterning* methods and other approaches such as *scanning probe lithography*, *nanoimprint lithography* and *self-assembly* show promise for precise manufacturing of nanoelectronic devices.

6. Looking ahead, advances and challenges

This chapter has reviewed recent developments in nanoscale architectures for smart bio-interfaces, with focus on four main areas: (i)the modeling and simulation to enable

nanofabrication by-design; (ii)the organic-inorganic, hybrid, conductive polymer-based nanomaterials for enhanced sensing, actuation; (iii)the lamellar plasmonic nano-interfaces for optical sensors; (iv)the controlled molecular functional architectures for thin film transistors. The specific nanofabrication methods and results were emphasized in each case and a number of references were selected.

The National Nanotechnology Initiative (NNI) was created in the United States in the year 2000 (Roco, Williams & Alivisatos, 1999). A recent peered-reviewed study, so-called *Nano2 Report,* was prompted by a decade of NNI mandated-efforts (www.wtec.org/nano2). This report "Nanotechnology Research Directions for Societal Needs in 2020"(Roco, Mirkin & Hersam, 2010) aptly highlights the main advances since the year 2000, the fundamental goals by 2020 and the expected challenges to be overcome. Interestingly, the research directions reviewed include "theory, modeling & simulation", "nanoelectronics & nanomagnetics", "nanophotonics & plasmonics", with brief conclusions in an easy to follow, table format. Thus, we would refer the reader to this report, due to text space constraints.

In terms of dealing with risk and benefits of nanofabrication and nanotechnology, there are two extreme views, with everything else in between. While the "optimistic" side emphasizes mostly the colossal potential benefits, the "pessimists" are accentuating the major risks involved, this occasionally heating up as a public policy debate.

In fact that, today, nanofabrication-enabled products are marketed to an estimated *254 thousand million* US dollars (USD) worldwide, with *91 thousand million* USD in the United States alone. These global trends foretell a doubling, every three years, of the number of nanofabricated products and dedicated workers. It seems that the nanotech-fueled optimism of Ratner & Ratner is carried along: experts are now projecting for 2020 a 3 trillion USD market with 6 million workers involved. We would all wish to get some of that, would we not? Perhaps this optimistic view of nanofabrication and nanotechnology would be the best way to conclude.

7. Acknowledgements

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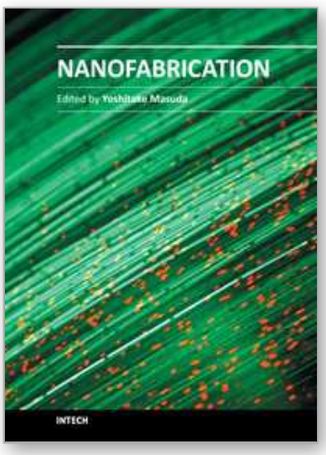

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We face many challenges in the 21st century, such as sustainably meeting the world's growing demand for energy and consumer goods. I believe that new developments in science and technology will help solve many of these problems. Nanofabrication is one of the keys to the development of novel materials, devices and systems. Precise control of nanomaterials, nanostructures, nanodevices and their performances is essential for future innovations in technology. The book "Nanofabrication" provides the latest research developments in nanofabrication of organic and inorganic materials, biomaterials and hybrid materials. I hope that "Nanofabrication" will contribute to creating a brighter future for the next generation.

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