

# **INVITED LECTURES**



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## **THE POTENTIAL OF NANOMATERIALS FOR HYDROGEN STORAGE: PROGRESS, PROBLEMS AND PERSPECTIVES**

Hydrogen is considered as one of the alternative solutions regarding the problem of rapidly shrinking fossil fuels and increasing greenhouse gasses levels. Being environmentally friendly, it has advantages like high energy density and high elemental abundance. However, the major bottleneck for the potential application is the lack of safe, economical and efficient on-board hydrogen-storage since none of current technologies can fulfill the main targets set by US Department of Energy.

Hydrogen-storage methods can be in general classified into physical and chemical methods. The challenge is to obtain material for on-board applications which will show a dual nature of physisorbed and chemisorbed material with the hydrogen binding energy about 15–30 kJ mol<sup>-1</sup>, that is strong enough to hold hydrogen but weak enough for fast hydrogen release [1]. Such a material has to meet also several other important challenges like fast kinetics, high gravimetric and volumetric storage capacity, manipulation should be possible in near ambient conditions regarding temperature and pressure, etc.

Hydrides such as MgH<sub>2</sub>, LiH, NaAlH<sub>4</sub>, LiBH<sub>4</sub>, etc. are among the most studied solid-state hydrogen-storage materials because they are relatively cheap, lightweight and have high storage capacity. Drawbacks such as too high (de-)hydrogenation temperatures, too slow kinetics and problems with reversibility limit their practical on-board automobile applications.

However, some potential solutions have been already proposed. For instance, theoretical studies suggest that selected metal intercalated carbons are expected to lower significantly the thermodynamic stability of the respective hydrides and show a dual nature [2]. These are expected to lower thermal stability but also have faster kinetics, remarkably enhanced molecular H<sub>2</sub> adsorption. Other theoretical concept focusses on the nano-sizing aspect; particularly tremendous change of the enthalpy of formation should be observed in various particles with the size below 5 nm [3].

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## **RECENT ADVANCES IN SYNTHESIS AND APPLICATIONS OF CARBON-ENCAPSULATED MAGNETIC NANOPARTICLES**

The unique size-dependent properties of magnetic nanoparticles attract the attention of physicists, chemists and engineers. This is reflected in the numerous prospective applications of magnetic nanoparticles, which include data storage, magnetic sensors, mobile catalyst platforms, environmental protection and various biomedical challenges (drug delivery, hyperthermia, targeted molecular imaging). The nanoparticles of non-oxide magnetic phases (e.g. transition metals) have substantially better magnetic characteristics in comparison to their oxide counterparts, especially very commonly studied iron oxide nanoparticles. It can be expressed in the values of saturation magnetization, which achieves 220 emu/g and 80-90 emu/g for metallic iron and iron oxide, respectively. The magnetic nanoparticles made of pure metallic phases, despite better magnetic performance, may undergo spontaneous unwanted and uncontrollable reactions: (i) surface oxidation, (ii) agglomeration, and (iii) corrosion. The specific properties of pure metallic phase magnetic nanoparticles can be preserved by encapsulating them into thin protective coatings. Carbon is the best protective agents, since it has (i) low density, (ii) high resistance to acids, bases and greases, (iii) high thermal stability. Moreover, carbon coatings are readily susceptible to functionalization processes, which can be applied for controlled and predictable surface modification of carbon-encapsulated magnetic nanoparticles (CEMNPs).

This work summarizes the recent advances in synthesis and application of carbon-encapsulated magnetic nanoparticles. The special attention is paid on the critical evaluation of yield and selectivity of typical methods that are routinely used for fabrication of CEMNPs, i.e. carbon-arc discharge, flowing thermal plasma, chemical and laser-assisted vapor deposition, combustion synthesis and hydrothermal synthesis. The prospective application of CEMNPs are thoroughly discussed in the frames of their suitability in sorption of heavy metal ions and organic compounds, degradation of environmentally toxic compounds, and molecular imaging of cancer tumors.

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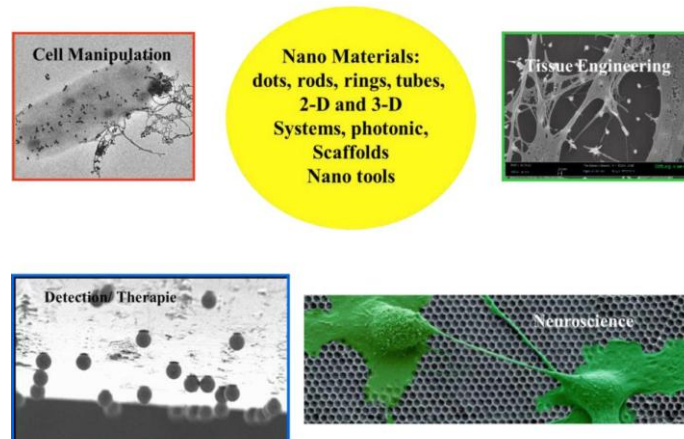
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## MULTIFUNCTIONAL NANOMATERIALS AND THEIR APPLICATION IN THE FIELD OF BIOMEDICINE

Nanotechnology applied to biology requires a thorough understanding of how molecules, sub-cellular entities, cells, tissues and organs function and how they are structured. The merging of nanomaterials and life science into hybrids of controlled organizations and functions is possible, assuming that “biology is nanostructured”, and therefore man-made nano-materials can structurally mimic nature and complement each other. By taking advantage of their special properties, nanomaterials can stimulate, respond to and interact with target cells and tissues in controlled ways to induce desired physiological responses with a minimum of undesirable effects. To fulfil this goal, the fabrication of nano-engineered materials and devices has to consider the design of natural systems. Thus, engineered micro-nano-featured systems can be applied to biology and biomedicine to enable new functionalities and new devices. Amongst others these include nanostructured implants providing many advantages over existing, conventional ones, nanodevices for cell manipulation, and nanosensors that would provide reliable information on biological processes and functions.

### Nanomaterials for application in life sciences



Cells: human; stem; cancer; fibroblast; osteoblast; fetal; bacterium; yeast; plants

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## **PROJECT NANOFORCE**

Nanoscience and nanotechnology represents new approaches to research and development (R&D) that concern the study of phenomena and manipulation of materials at atomic, molecular and macromolecular scales. NANOTECHNOLOGY currently underpins many practical applications (medical, ICT, energy production, food-water, security, a broad range of materials, etc.) and has the potential to enhance quality of life and environmental protection, and boost industrial competitiveness.

Along with seven European partners, Chemie-Cluster Bayern has started the EU-funded INTERREG project NANOFORCE. NANOFORCE strives to foster innovative nanotechnology sector networks across Central Europe regions by bringing together enterprises, research centers, venture capitalists and public institutions to carry out collaborative & interdisciplinary research projects on nanomaterials (in the frame of REACH Regulation) and to turn the most promising laboratory results into innovative industrial applications - not only to produce new materials, but also to improve industrial sustainability (more security & lower environmental impact in the product life cycles). 100 “Nano-Deals” involving operators from the chemical sector are expected to be generated until the end of the project lifetime. Implementation of joint ventures initiated within the scope of NANOFORCE will be supported through an interregional Venture-Capital Fund.

The project covers

- Framework analysis to outline the current situation of the nanotechnology sector and gather information about the innovative level and gained experience in nanotechnology of chemical companies and governance authorities
- Identification of potential needs and gaps in legislations and risk assessment and collection of data to establish baseline reports for specific target groups
- Laboratory analysis and exposure scenarios establishment on 3 major nanomaterials
- Putting down State-of-the-art of regulations in the field of nanotechnology to identify needs and gaps and give recommendations for the European Commission
- Information dissemination during project workshops for target groups in the nanotechnology sector to outline the project objectives and results
- A nano-deal generator platform established to create potential deals between R & D and industry and to create transnational joint ventures in the nanotechnology sector

Within this contribution the main activities of the NANOFORCE project will be presented in detail as well as the first results achieved.

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## **DO IT IN WATER! AQUEOUS (CO)PRECIPITATION PROCESSES FOR NANOPARTICLES AND NANOSTRUCTURED PARTICLES**

At the time being, a gap is increasing between lab-scale nanomaterials syntheses, searching for originality regardless of industrial scale-up, and acceptable processes for mass-production. Even if non-aqueous chemistry is able to produce fascinating and complex nanoparticles at lab scale, aqueous chemistry is much more relevant to meet industrial concern about costs, safety and environment.

We will show some examples illustrating how particle morphology and nanostructure can be controlled in aqueous systems, playing with physico-chemical and process parameters in reactors allowing easy scale-up. A special attention will be given to systems exhibiting morphological transitions vs pH or temperature, including transitions from isolated nanoparticles to multi-scale particles (e.g. nickel hydroxide, zinc oxide). We will also give an overview of some interesting nanoheterostructures obtained in aqueous systems.

We will finally tackle the problem of mixed (hydr)oxides coprecipitation, including the case of cations with very different solubilities, and discuss the main strategies (e.g. alkaline hydrothermal crystallization).

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## NANOCRYSTALLINE STATES OF PAULI PARAMAGNET

The  $\text{YCo}_2$  intermetallic compound is a Pauli exchange-enhanced paramagnet. In a fine crystalline form it becomes magnetically ordered in the whole volume of the grains. Also in amorphous state the ferromagnetism appears. In our studies the magnetic properties of rapidly quenched  $\text{YCo}_2$ -type compositions are described for the nanocrystalline state produced by rapid quenching at an appropriate cooling rates.

The intermetallic compounds  $\text{YCo}_2$ ,  $\text{Y}_{0.9}\text{Nb}_{0.1}\text{Co}_2$  and  $\text{Y}_{0.9}\text{Ti}_{0.1}\text{Co}_2$  studied were examined with X-ray diffraction (XRD) and vibrating sample magnetometry (VSM). The samples with up to 10 at.% substitution consist of a single phase which grows after partial replacement of Y atoms in the initial  $\text{YCo}_2$  by Nb or Ti.

The electronic band structures of  $\text{YCo}_2$  systems with point defects were calculated using FP-LAPW (Full Potential–Linearized Augmented Plane Wave) method. Nb and Ti substitutions and also vacancies were considered as a point defects. Theoretically predicted ferrimagnetic ground states for the  $\text{YCo}_2$  system with Y atoms replaced by Ti and Nb find experimental confirmation in  $\text{Y}_{0.9}\text{Ti}_{0.1}\text{Co}_2$  and  $\text{Y}_{0.9}\text{Nb}_{0.1}\text{Co}_2$ . Calculations show that ferrimagnetic ground states are energetically more stable for systems with point defects at the sites of Y atoms than at the sites of Co ones. Reduction in the magnetic moment values of Co to about  $1 \mu_B$  for compounds with point defects at cobalt sites is found.

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## MICROWAVE SOLVOTHERMAL SYNTHESIS OF NANOPARTICLES

We will present a series of reactors for synthesis of nanoparticles and other chemical reactions in Solvothermal conditions, with microwaves as energy source. Hydrothermal synthesis is a wet chemical process where the reagents are closed in a high pressure vessel, and as temperature rises, it becomes higher than the boiling point at atmospheric pressure. When the process is carried in non-aqueous solvent, it is called Solvothermal. Many reports have shown its considerable advantages when synthesis of nanoparticles is concerned, first of all high crystallinity of the particles.

Further improvement can be achieved by applying microwaves to heat the reagents. The process, which we call Microwave Solvothermal Process, permits to precisely control the reaction time, and to heat the reagents with a rate of above 1 K/s, due to the high power density of over 5 mW/ml transmitted to the reagents with small temperature gradients only. A main barrier for application of this technology was lack of suitable reactors, with good control of the process, high efficiency, and throughput.

We present a series of reactors: the Ertec – Magnum reactor for batch synthesis, with which we performed our first synthesis at pressures till 10 MPa and 300°C for 150 ml batches, MSS-1: a continuous flow or stop/flow reactor working under pressures up to 4 MPa, MSS-2: a stop-flow reactor for pressures up to 6 MPa, and chamber volume 400 ml, and MSS-#, a reactor for MSS synthesis at pressures up to 20 MPa.

We will also show a product: nano-hydroxyapatite (HAP) where the nano-particle size, stoichiometry, and solubility rate can be precisely controlled by varying the synthesis time from 90 sec to 6000 sec, and the rapid temperature ramp of 1K/sec at the reaction start as well as precise reaction time control is crucial to obtain a highly biocompatible material for bone regrowth applications. The nano-material has been registered in the patent office as GoHAP.

We can also offer for laboratory tests and collaboration in development of new technologies nano-ZnO, ZnO alloyed with Co, Mn, Mg, and other ions, as well as Zirconia nanoparticles doped with rare earth ions or Calcium.

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## **FOURIER TRANSFORMED INFRARED SPECTROSCOPY IN THE MID AND LONG WAVELENGTH SPECTRAL RANGE**

Modulation spectroscopy is an excellent tool to study the energies of optical transitions (including the excited state ones) in low dimensional semiconductor systems. Such experiments have already been successfully employed to study GaSb-based QWs for infrared applications up to 3  $\mu\text{m}$  [1] and including type II structures for even longer wavelengths [2]. Because there is a growing interest and necessity to investigate structures designed for operation in mid and far infrared range (e.g. infrared detectors, lasers etc.) it has been proposed to exploit modulation spectroscopy realized by using Fourier transformed spectrometer [3]. In this work, it will be demonstrate the application of FTIR modulation spectroscopy for investigation of low-dimensional structures like type I or II quantum wells designed for mid infrared spectral region (up to  $\sim 7 \mu\text{m}$ )[3]. In addition, the Fast Differential Reflectance (FDR) spectroscopy will be introduce[4,5]. This technique allow to posses the photoreflectance-like spectra similar to this achieved by modulation concept utilize lock-in amplification technique. Nevertheless in FDR approach, the time of collecting spectra might by reduce from hour to seconds in comparison to standard grating- based technique. In this part, the results obtained for the materials being results of infrared gas sensing requirements like e.g. GaInAsSb/InAs layers [6] and type II quantum wells [7] will be present. Finally, future plans of optical studies, realize within this setup, are going to be discussed.

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## **NANOPHYSICS CLOSE TO THE ULTIMATE LIMIT**

The transition from classical to quantum physics can be easily reached in nanostructures. However, for quantum effects to be still dominant at room temperature typical sizes of only a few nanometers are required, with the ultimate limit being at single molecules as functional units. The quantization dominant at these small length scales offers new possibilities for technology, but, on the other hand, requires first the understanding of new and very fascinating physical concepts governing such structures. Furthermore, these structures are low-dimensional ranging from two dimensions (2D) in graphene sheets or in surface states to one dimension (1D) in atomic wires and finally to zero dimensions in clusters and molecules with specific properties depending on dimension, but also on their contact and interaction with a three-dimensional environment. As a result, specific electronic states confined at surfaces, interfaces or even at edges are observed with new peculiar properties that are for a certain class of materials even topologically protected.

I will discuss several examples ranging from 2D to 0D, concentrating on the correlation of local structure on the atomic scale with collective excitations (plasmons) and dc conductive properties. Emphasis will be on physical properties with some perspectives for technical applications.

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## **ION-ION INTERACTION AS A TOOL FOR CONTROLLING SPECTRAL AND TEMPORAL PROPERTIES OF ULTRASMALL INORGANIC OPTICAL MARKERS BASED ON LANTHANIDES EMISSION FOR BIO-MEDICAL APPLICATIONS**

Introducing to medicine and biology concept of optical markers in tremendous way has changed the recent status of these two important disciplines. This was mainly due to strong development in imaging techniques which recently allow us to investigate both static as well dynamic properties of living cells, their components and their interactions with external factors. Recently used molecular markers including organic dyes, fluorescent proteins or chelates containing lanthanide ions have several significant limitations. One of the alternatives for molecular markers are inorganic quantum dots (ie. CdSe, CdS) which are recently commonly used in many academic works. However, even if they are much better from physico-chemical point of view, from the application point of view at this moment they are rather useless mainly because of their high risk of toxicity and difficulties in obtaining infrared excitation/emission. One of the solution combining advantages of both concepts is to make nontoxic inorganic nanocrystals doped by lanthanide ions.

In this work, we will present optical results obtained mainly for NaYF<sub>4</sub> and NaGdF<sub>4</sub> nanocrystals doped with different lanthanide ions (Eu, Tb, Er, Tm, Yb). The aim of this work was to design and to synthesize these markers and to understand physical processes responsible for their emission/excitation and to optimize these processes to the physical limits. In this aspect, it will be shown how the ion-ion interactions can be used to control the spectral and temporal properties of such nanocrystals [1, 2]. Moreover, the influence on optical properties of core-shell architecture [3], size, shape and surface of nanocrystals will be also discussed in details.

Additionally, the potential use of such nanocrystals in nano-bio medicine will be discussed. In this aspect, we will discuss preliminary results obtained for such nanocrystals in the field of immuno- and nephro- toxicity. Moreover, different aspects of nanocrystals surface functionalization will be also discussed.

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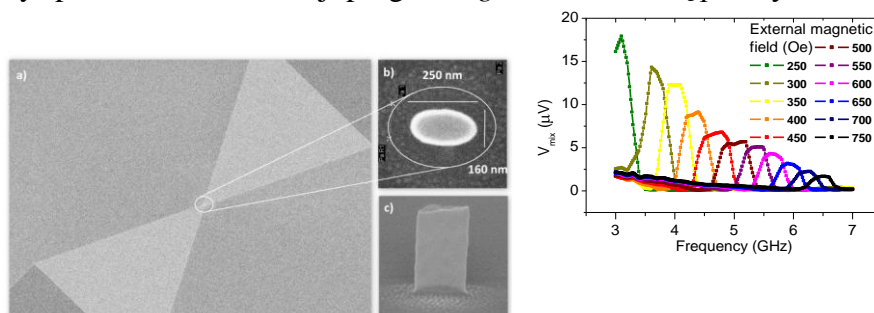
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## CO NOWEGO W SPINTRONICE?

Od czasu pionierskiej pracy Julliere (1975) wykazującej w temperaturze pokojowej tunelowanie elektronów pomiędzy ferromagnetycznymi elektrodami (tunelowa magneto-rezystancja  $TMR=2.7\%$ ), w chwili obecnej, magnetyczne nano-złącza tunelowe z cienką, sterowaną barierą  $MgO(001)$  wykazują  $TMR=604\%$  [1]. Dzięki tak dużemu przyrostowi magneto-rezystancji, nano-złącza TMR są stosowane jako elementy odczytowe w głowicach nowej generacji twardych dysków o wysokiej gęstości upakowania informacji ( $> 500$  Gbit/cal<sup>2</sup>), jako komórki pamięci M-RAM, ST-RAM i mikrofalowe oscylatory precesji spinowej. Firma EverSpin w tym roku wprowadziła na rynek 64 Mbit, nie wymagającą odświeżania, pamięć ST-RAM.

W referacie zostanie przedyskutowane zjawisko spinowego transferu momentu siły (Spin Transfer Torque, STT), polegające na przełączaniu magnetyzacji elektrody detekcyjnej złącza za pomocą prądu spinowo spolaryzowanych elektronów, w zastosowaniu do wytworzonych w Katedrze Elektroniki AGH, komórek pamięci ST-RAM [2,3] i Spin Torque oscylatorów (rys. 1) [4]. Drugi temat, obecnie intensywnie badany w wielu laboratoriach elektroniki spinowej na świecie jaki zostanie omówiony, to modyfikacja własności statycznych (anizotropii magnetycznej) [5] i dynamicznych (precesja spinowa i przełączanie magnetyzacji) za pomocą pola elektrycznego przyłożonego do złącza. W podsumowaniu, perspektywy spintroniki w realizacji programu *green IT* zostaną przedyskutowane.



Rys. 1. Przykładowe nano-złącza tunelowe  $FeCoB/MgO/FeCoB$  wraz z widmem ST-FMR

Badania są finansowane w projekcie NANOSPIN PSB-045/2010 przez Szwajcarię w ramach szwajcarskiego programu współpracy z nowymi krajami członkowskimi Unii Europejskiej.

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## NANOSECOND AND SUB-NANOSECOND TIME-RESOLVED SPECTROSCOPY OF LOW-DIMENSIONAL QUANTUM STRUCTURES

Time-resolved spectroscopy is of great importance for current research concerning dynamics of charge and spin degrees of freedom of confined particles in various systems, including low dimensional semiconductor structures. Information about kinetics of electronic phenomena in solid state and related physical processes gives important contribution to the process of designing and developing of modern devices, in which quantum phenomena are crucial or determine the device functionality. Often, the analysis of experimental data originating from the time-domain experiment provides information unavailable in other types of experiments. The dynamic change of the electronic structure of the investigated system [1,2], the regime of charge and spin confinement [3], the coupling between two low-dimensional systems [4,5] are only a few of existing examples.

In my talk I will present the modern and most sophisticated approach concerning measurement of charge and band structure dynamics in the nano- and subnanosecond time scale. I will focus on two types of quantum structures: (i) structures with a spatial separation of electrons and holes, and (ii) new class of asymmetric quazi-zero dimensional quantum objects – quantum dashes. In the case of structures with spatial charge separation I will discuss the layered system with two-dimensional density of states as well as a quazi-zero dimensional system with self-assembled quantum dots. I will show essential advantages of applying the two-dimensional imaging of the quantum system band structure evolution in the streak camera measurement. In the case of the quantum dash (QDash) structure I will discuss the intra-band and inter-band carrier relaxation and describe the regime of quantum confinement which depends strongly on the QDash size. Taking into account the wavelength of QDash related emission, which falls into 1.3-2.0  $\mu\text{m}$  range, information about carrier dynamics in this system was hardly accessible by a more standard approach. I will show that thanks to the application of the photon counting technique with superconducting detectors, a new type of a streak camera system and the pump-probe experiment it is possible to provide a wide spectrum of data related to charge dynamics and regime of quantum confinement in this new class of quantum objects.

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## **GRAPHENE NANORIBBONS ON SOLID SURFACES**

Possibility of controlled synthesis of covalently bond hydrocarbon chains on solid surfaces opens new opportunities for “bottom-up” fabrication of graphene nanoribbons, i.e., stripes of single-layer of graphite with semiconducting properties. So far, the only successful experiments have been performed at single crystal surfaces of noble metals (such as Au(111) and Ag(111)), the substrates which do not provide electronic decoupling of the graphene structures, necessary for their characterisation and prospective applications in electronic devices. In my talk, I shall review the “state-of-the art” in graphene nanoribbon research on metal substrates and report on recent results obtained in my group on controlled self-assembling and polymerisation of selected halogenated anthracene molecules taking place on semiconducting surfaces of rutile  $\text{TiO}_2$  and passivated Ge:H. It will be demonstrated for the first time, that long covalently bond chains of polyanthrylenes could be successfully constructed on rutile surfaces and several scenarios for thermally activated cyclo-dehydrogenation processes leading to nanoribbon formation will be discussed and illustrated with the atomically resolved experimental STM images obtained “in situ” at cryogenic temperatures.

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**SELECTED EXAMPLES FOR APPLICATIONS, PROPERTIES AND  
ECONOMICAL ASPECTS OF NANOTECHNOLOGIES WITH A FOCUS ON  
CARBON BASED MATERIALS**

Nanotechnology and C-based materials are gaining more and more interest even in the financial community. From a commercial point of view nanomaterials and nanotools - that cover for example analytical instruments and to some extent, equipment for the semiconductor industry are already in the market or have the best opportunity to be commercialized soon.

At the beginning of the value chain are clearly materials. A good example currently for rapid development are carbon nanotubes. The success or failure of carbon nanotubes depends on their market applications. Carbon nanotubes could play a pivotal role in the upcoming nanotechnology age if their remarkable electrical and mechanical properties can be successfully exploited in products. Nanotubes offer significant advantages over most existing materials such as carbon fibers, including: an unimagined strength-to-weight ratio; attractive mechanical properties; and very interesting field emission characteristics. Mass market applications may include reinforcement of polymer nanocomposites and field emission displays. Nanotube molecules might line up to form a light, strong wire or a composite that could be unbeatable as a material for making lightweight vehicles for space, air and ground. However, recent developments herald a technology breakthrough which could allow mass production of carbon nanotubes. Several predictions indicate that the global production increases from the few kilogram range to more than 100 tons per year coupled with an expected significant price reduction. Another interesting and upcoming material for niche market applications is nanodiamond. Several applications for nanodiamond will be introduced in this talk.

In general, the term nanotechnology is badly defined and useful market figures – with a few exceptions – do not exist so far. One interesting area is the border between microsystems and nanotechnology. In certain microsystems applications – e.g. for hard disk drive heads based on giant magnetoresistance (GMR) read heads – nanotechnology already plays a significant role in a multi-billion dollar market. GMR-sensors in automotive applications also play an important role for next generation products. For other microsystems applications like polymer displays the impact of nanotechnology is developing rapidly.

In this presentation, some market figures and applications in the nanotechnology area will be discussed. Selected nanotechnology companies as well as their products will be presented from a market perspective application point of view. A special focus of the talk will be on the nanomaterials sector and nanotechnology applications in different areas.



*oxide matrix, rare-earth ions, luminescent nanomarkers,  
solvothetical technology, hydrothermal technology, early recognition,  
cancer detection, microwave, ZrO<sub>2</sub>, ZnO, ZnAl<sub>2</sub>O<sub>4</sub>*

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### **PERSPECTIVE LUMINESCENT NANOMARKERS BASED ON RARE-EARTH DOPED OXIDE MATRIXES: FROM TECHNOLOGY TO APPLICATION**

Since 80's to nowadays one can observe the intensive research over the new diagnostic methods, allowing early recognition and effective therapy of different types of cancers. After analyzing year after year improving cancer statistics, the correlation between early cancers diagnostic and recovery rates is confirmed. At an early stage of disease the therapy is less invasive or even non-invasive. Early cancer diagnosis methods should be non invasive or less invasive, compared to the ones used at present. One of the most promising method for early diagnosis and even treatment of cancer is based on achievements of the nanotechnology and the use of nanoparticles, nanopowders and luminescent nanomarkers. The application of the luminescent nanomarkers allows detection of cancer tumors at early stage. Luminescent nanomaterials (nanomarkers) based on wide band gap semiconductor compounds doped with rare-earth ions are good candidates for early cancer diagnostics tool. Nanomarkers based on different oxide matrixes will be shown. The possibilities of the markers parameters control will be discussed, since the certain properties are necessary for their medical and biological applications. The optical properties of luminescent materials will be analysed. Biomedical results indicate the usability of the studied materials as perspective luminescent nanomarkers, in particular for the use for early recognition.

