

Second NIMS-WUT-EMPA Workshop Materials for Sustainable Development November 12th – 13th, 2009

Venue:

National Institute for Materials Science, Namiki-site (Collaborative Research Building, Large Meeting Room 403)

List of Abstracts

- NIMS National Institute for Materials Science, Tsukuba, Japan
- WUT Warsaw University of Technology, Poland
- EMPA Swiss Federal Laboratories for Materials Testing and Research, Switzerland

Oral Presentation 1-1

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Home Page:

Presentation Title:

Modelling based engineering of grain boundaries in metals

Abstract:

The paper presents the results of multi-scale modelling of the grain boundaries in metals and alloys and their effect on the properties of polycrystalline engineering materials. Atomistic simulations are used to predict the properties of individual grain boundaries with special attention paid to their energy, which determines the resistance to corrosion and fracture strength and other properties.

For multi-grain structures, Monte Carlo methods are employed for modelling the migration of grain boundaries during grain growth. Different grain growth paths are simulated and the thermal stability of fine grained polycrystals is analyzed. At the same length scale, the theory of graphs has been used to simulate the propagation of the grain boundary cracks and the fracture resistance of polycrystalline aggregates.

The effect of grain boundaries on the macroscopic properties of nano-polycrystalline metals is modelled by Finite Element Method, FEM. In particular, FEM has been used to analyse the effect of residual stresses on the yield and flow stress of nano-grained metals.

Finally, the results of modelling at various length scales are discussed with respect to the possibility for the grain boundary engineering of nano-metals and alloys.



Oral Presentation 1-2

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Presentation Title:

Hydrogen storage: a materials science challenge for energy

Abstract:

The world wide energy demand increases just as rapidly as the average temperature of the atmosphere. The reserves of fossil fuels worldwide are limited and the combustion of the carbon fuels leads to a severe increase of the CO_2 concentration in the atmosphere. The latter is responsible for the climate change. The future of the industrialized word, i.e. the economy as well as the society, is determined by the ability to change from fossil fuels as energy carriers to renewable energy. The main difference between the fossil period and the future is the requirement of producing synthetic energy carriers. Hydrogen as an energy carrier opens the path to a society based on renewable energy.

The storage of hydrogen in metals and complex hydrides as stable compounds offers a great volumetric storage density. Metallic hydrides reach a volumetric hydrogen density of 115 kg·m⁻³ e.g. LaNi₅. Most metallic hydrides absorb hydrogen up to a hydrogen to metal ratio of H/M = 2. Greater ratios up to H/M = 4.5 e.g. BaReH₉, have been found [2], however all hydrides with a hydrogen to metal ratio of more than 2 are ionic or covalent compounds. The highest volumetric hydrogen density known today is 150 kg·m⁻³ found in Mg₂FeH₆ and Al[BH₄]₃, both hydrides belong to the complex hydrides. However the gravimetric storage density is limited to less than 20mass% in the materials.

In order to replace fossil fuels without scarification on energy density synthetic fuels based on hydrogen e.g. NH_3 or C_8H_{18} have to be developed. The latter also represents a effective CO_2 sink for the atmosphere.

Reference:

[1] Züttel A, "Hydrogen Storage Methods", Naturwissenschaften 91 (2003), pp. 157-172



Oral Presentation 1-3

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Presentation Title:

Tailoring nanostructured oxide thin films for the miniaturization

of solid oxide fuel cells

Abstract:

The development of micro-solid oxide fuel cells (SOFCs) open new scenarios for portable unit power generation. Micro-SOFCs are likely to produce energy densities per volume and specific energy per weight up to four times larger than state-of-the-art batteries [1]. Reducing the operation temperature is critical for practical use of miniaturized SOFCs and can be achieved using thin-film electrolytes [2]. However, lowering the SOFC operating temperature makes slow the kinetics of charge transfer reactions at the electrolyte-electrode interfaces, mainly at the cathode. This results in a worst fuel cell performance, because the lower the fuel cell working temperature, the larger the cathode overpotential. An increase in their triple boundary phase (TPB) can improve electrode performance.

Pulsed laser deposition (PLD) is very promising for the fabrication of tailored oxide thin films, because it allows to obtain single crystal or polycrystalline films, with tailored microstructure from dense to highly porous, as we demonstrated in previous works [3]. SOFC components

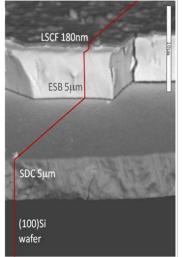


Fig. 1 Tri-layer of LSCF cathode, ESB and SDC electrolyte films, deposited by PLD on (100) Si wafer.

and their assembly, such as dense oxygen ion (ceria), bilayer (ceria/bismuth oxide) and protonic (barium zirconate) electrolyte films in single crystal and polycrystalline forms were fabricated by PLD, with cathode films (LSCF perovskite oxides) deposited on top (Figure 1). Aim of the MANA research project is the fabrication of micro-SOFC devices, starting from the understanding of the nanostructured materials performance as single components (electrolytes) or assembled with the electrodes.

References:

[1] A. Bieberle-Hütter et al., J. Power Sources Vol. 177 (2008) 123.

- [2] S.J. Litzelman, J.L. Hertz, W. Jung, H.L. Tuller, Fuel Cells Vol. 5 (2008) 294.
- [3] D. Pergolesi et al, ECS Trans. Vol. 7 [1] (2007) 891.

Oral Presentation 1-4

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Presentation Title:

Nanostructured AgCu brazing filler in a multilayer configuration

Abstract:

Too high brazing and soldering temperatures limit the joining of advanced alloys. The nanoeffect melting point depression allows to lower the melting temperature of the filler metal without the compromise known from the alloying approach. The nanomultilayer configuration, where layers of the filler metals alternate with Diffusion Barrier Layers (DBL), is one way to apply a nanostructured filler metal.

Melting point depression of 230°C was observed for the 2.3nm thick AlSi nanolayers in a AlSi/AlN nanomultilayer configuration. The melting behaviour of AgCu in nanomultilayer configuration depended on the DBL applied. Tungsten and Carbon as DBL exhibited similar melting point depression behaviour and a eutectic melting character. AlN as DBL has shown less of the melting point depression, and a non eutectic melting behaviour. The Ag phase was partially solid at temperatures even higher than the AgCu bulk melting point.

The interface between Ag and AIN strongly influences the melting behaviour. Compared to other DBL the Ag-AIN interface shows the smallest lattice misfit (5-6%) and a similar crystal structure at the interface which results in an epitaxial growth of Ag on AIN. The semi-coherent and energetically favourable Ag-AIN interface changes the eutectic composition of the AgCu nanolayers. This work shows that phase diagrams of nanostructured materials are additional influenced by the interface character.

Reference:

Patent: DE102008050433.5 Inventors: Vinzenz Bissig, Mathias Türpe, Peter Englert, Jolanta Janczak-Rusch.



Oral Presentation 1-5

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Presentation Title:

Crystal growth of ZnO thin films on flexible polymer substrates at room temperature by introducing nanosheet seed layer

Abstract:

Thin film technology is one of the most basic technologies used to produce various devices which underpin today's information-intensive society, and thus the design and orientation control of the crystal films on inexpensive substrates such as glass or plastic have been extensively studied. Recently, we reported a novel method to control the orientation of sol-gel deposited films by using unique two-dimensional (2D) crystals called "nanosheet"^[1] as a seed. In this study, we tried to apply this technique to vacuum deposition method to realize well-crystallized and oriented growth of functional oxide films on plastic substrates.

Highly organized monolayer films of nanosheets were produced onto glass and polymer (PET: polyethylene terephthalate, PEN: polyethylene naphthalat) substrates via Langmuir-Blodgett deposition. $Cs_4W_{11}O_{36}$ nanosheet (with 2D-hexagonal lattice) was used as a seed, which was synthesized by delamination of $Cs_{6+x}W_{11}O_{36}$ layered host oxide. ZnO films were fabricated onto the substrates at room temperature by using pulsed laser deposition (PLD). Characterizations by XRD and TEM revealed that the deposited films were highly crystallized, and oriented along the *c*-axis. Highly crystalline texture directly formed on top of the substrates has been realized for the first time. The results suggested that the growth of ZnO crystal can be critically controlled by the top-most structure of the nanosheet regardless of the substrate species (glass, plastics). Drastic improvement of film performance was successfully demonstrated by measuring the electrical conductivity of the films.

Reference:

[1] T. Shibata et al., Adv. Mater. 20, 231 (2008).



Oral Presentation 2-1

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Presentation Title:

Detection of biomolecular recognition using bio-transistors

Abstract:

We have proposed the novel concept of a genetic field effect transistor (FET) which is based on direct transduction of surface density change of charged biomolecules into electrical signal by the field effect¹. We combined a genetic FET with specific molecular recognition such as allele specific hybridization, intercalation, and primer extension to detect one base difference of target DNA.

The conceptual structure of a genetic FET is shown in Fig. 1. Oligonucleotide probes are immobilized on the surface of the gate insulator. The genetic FET is immersed in a measurement solution together with an Ag/AgCl reference electrode with saturated KCl solution. The photograph of the fabricated FET chip is shown in Fig. 2.

The cycle of single-base extension and measurement of the V_T was repeated iteratively to determine the base sequence of the target DNA. As a result, the positive V_T shifts could be detected in accordance with V_a

the base sequence of the target DNA. Thus, the results of iterative extension reaction and detection of the threshold voltage indicated the ability of a direct, simple and potentially high throughput DNA sequencing analysis using the FETs.

We also propose an oocyte-based field effect transistor (oocyte-based FET) for drug analysis. which transport in target transporters are expressed at the cell membrane of the oocyte. Non-invasive monitoring of the uptake kinetics of substrates mediated by membrane-bound realized transporters can be with oocyte-based FET.

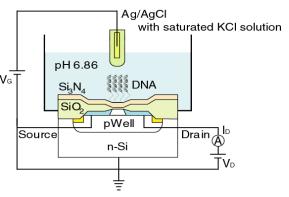


Figure 1 Schematic diagram for measurements of electrical characteristics of genetic FET.

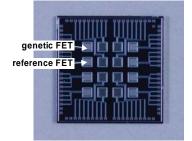


Figure 2 Photograph of the fabricated genetic FET chip. Sixteen FETs and a temperature sensor are integrated in a 5 mm x 5 mm chip.

Reference:

1) T. Sakata and Y. Miyahara, DNA sequencing based on intrinsic molecular charges, Angewandte Chemie International Edition, 45, 2225-2228 (2006)



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Presentation Title:

Boron nitride nanotubes as innovative biomaterials

Abstract:

Boron nitride nanotubes (BNNTs) are of significant interest for the scientific community because of their potentially unique and important properties for structural and electronic applications¹. A BNNT is a structural analogue of a carbon nanotube (CNT) in nature: alternating B and N atoms entirely substitute for C atoms in a graphitic like sheet, with almost no change in atomic spacing; despite this, carbon and boron nitride nanotubes present many different properties. While BNNTs possess a very high Young's modulus similar to CNTs, they have superior chemical and thermal stability and, contrary to CNTs, a constant band gap of about 5.5 eV. Recently, it has been moreover shown that BNNTs have excellent piezoelectric properties, superior to those of piezoelectric polymers². All these properties make BNNTs potentially attractive candidates for a wide range of applications in the nano-domain. In the last years, applications of CNTs in the field of biotechnology have been largely proposed³, but biomedical applications of BNNTs are totally unexplored yet.

In this presentation, we show our first studies on biocompatibility and interactions between BNNTs and living cells. Thereafter, we will introduce some potential useful applications of BNNTs in biomedical field, that range from drug delivery systems⁴, boron carriers for boron neutron capture therapy⁵, and to cellular nanotransducers⁶. Collectively, these experimental observations confirm that BNNTs are suitable for the development of novel nanovectors for cell therapy, drug, and gene delivery, and indeed for other biological and clinical applications⁷.

- ¹ Terrones M, Romo-Herrera J M, Cruz-Silva E, Lopez-Urias F, Munoz-Sandoval E, Velazquez-Salazar J J, Terrones H, Bando Y and Golberg D 2007 Pure and doped boron nitride nanotubes. Mater. Today 10 30.
- ² Nakhmanson S M, Calzolari A, Meunier V, Bernholc J and Buongiorno Nardelli M 2003 Spontaneous polarization and piezoelectricity in boron nitride nanotubes. Phys. Rev. 67 235406
- ³ Lacerda L, Bianco A, Prato M, Kostarelos K 2006 Carbon nanotubes as nanomedicines: from toxicology to pharmacology. Adv. Drug Deliv. Rev. 58 1460.
- ⁴ Ciofani G, Raffa V, Yu J, Chen Y, Obata Y, Takeoka S, Menciassi A, Cuschieri A 2009 Boron nitride nanotubes: a novel vector for targeted magnetic drug delivery. Curr. Nanosci. 5 33.
- ⁵ Ciofani G, Raffa V, Menciassi A, Cuschieri A 2009 Folate functionalised boron nitride nanotubes and their selective uptake by glioblastoma multiforme cells: implications for their use as boron carriers in clinical boron neutron capture therapy. Nanoscale Res. Lett. 4 113.
- ⁶ Raffa V, Ciofani G, Cuschieri A 2009 Enhanced low voltage cell electropermeabilization by boron nitride nanotubes. Nanotechnology 20 075104.
- ⁷ Ciofani G, Raffa V, Menciassi A, Cuschieri A 2009 Boron nitride nanotubes: an innovative tool for nanomedicine. Nano Today 4 8.



Oral Presentation 2-3

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Home Page:

Presentation Title:

Highly perfusive 3D scaffolds for thick tissue engineering

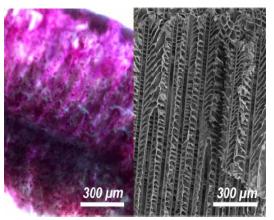
Abstract:

Tissue engineering (TE) and organogenesis have become a major focus of modern therapeutic approaches not only as an alternative to organ transplantation, but also as powerful instrument for the *in vitro* study of complex pathologies and physiological processes ^[1].

TE is based on the use of controlled three-dimensional constructs, usually denominated scaffolds, in which cells can be seeded before implantation and their growth directed to the constitution of living architectures functionally, structurally and mechanically equal to the tissue to be replaced ^[2].

To date, the lack of a vascular net and poor perfusion is what mostly prevents 3D scaffolds from being used in organ repair, when reconstruction of thick tissues is needed.

In the present work ^[3], scaffolds made of poly(L-lactic acid) (PLLA) with an overall porosity of 93% and interconnectivity over 90% were prepared by directional thermally induced phase separation (dTIPS) (figure right side). Mesenchymal (bone marrow stromal) stem cells (MSC) were vacuum-seeded down to scaffold thickness of 1 mm. Healthy, confluent proto-tissues were reconstructed after 14 days in vitro (figure left side) taking advantage of the highly-perfusive porous microstructure of the engineered scaffolds.



- [1] S. E. Place, D. N. Evans, M. M. Stevens, *Nature* **2009**, *8*, 457.
- [2] R. Langer, J. P. Vacanti, *Science* **1993**, *260*, 920.
- [3] C. Mandoli, B. Mecheri et al., *Macromol Biosci* **2009**, DOI.10.1002/mabi.200900323.

Oral Presentation 2-4

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Presentation Title:

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Nanojoining techniques and interface engineering

Abstract:

The rapid development of nanomaterials research will only prove useful when the materials synthesized, such as nanotubes, nanowires, nanostructured alloys and nanocomposites, can form integrated parts of devices and components. For instance, special joining techniques and methods are required to achieve nanoscale electronic devices from CNTs, to enable NEMS assembly, or to fabricate nanoscale sensing devices with molecular sensitivity. We define nanojoining as joining involving nanoscale joints or the joining of nanoscale materials. The industrial potential of nanojoining is significant as it is foreseen as an enabling technology for fields such as molecular electronics, conventional electronics and photonics, smart structures as well as structural applications.

The research activities of our group address the main challenges associated with the joining of nanomaterials. This involves studying the dependence of the most important joining attributes, such as wetting, adhesion, melting point of joint media on the length scale, the exploitation of mesoscale phenomena for new joining concepts, understanding the joining processes at the nanoscale to achieve controllable and consistent joints, and the development of novel mass production-friendly processes that do not destroy carefully designed nanostructures at the end of the value-added production chain.

More specifically, we are currently investigating the melting point suppression properties of nanoparticles to significantly reduce the joining temperature and to maximize thermal load during processes, the use of nanomaterials with higher reaction rates and shorter diffusion distances to reduce the processing time of joining processes, self-assembled joints using high surface area reactive nanoparticles, the use of nanocomposites multilayered materials to impart new functionalities to a joint, the improved adhesion properties of nanostructures to form robust temporary joints, and finally, the use of alternative energy sources for the joining of nanostructure-based electronic devices.

Reference:

S.S. Babu, S.Flowers, J.Janczak-Rusch, et.al., Joining of nanomaterials in Gennesys White Paper, Ed. H.Dorsch, M.H. Van de Voorde, MPI, Stuttgart, 2009

Oral Presentation 3-1

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Presentation Title:

Microstructural non-homogeneity of fine grained metals and its importance to their mechanical properties

Abstract:

Engineering materials are usually considered as homogenous. However, their microstructures, which consist of such elements as grains, grain boundaries, second-phase particles are inherently non-homogenous, when examined at sufficiently low-length scale. In the past, the non-homogeneity of metals was apparent at the scale of microns. With the recent advances in the refinement of metals structure well below 1 micron, the non-homogeneity is evident at nano-metric scale. This may have a profound effect on the mechanical properties of the nano-metallic materials. The paper demonstrates various examples of nano-structures in metals and describes methods and parameters for quantifying their non-homogeneity. The effect of these parameters on mechanical properties is evaluated experimentally by mechanical testing and modelled by numerical simulations.

Acknowledgement:

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Oral Presentation 3-2

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Presentation Title:

Development of ordered nanoporous materials for electrochemical applications

Abstract:

Mesoporous materials have received much attention due their large internal surface area and pore volume, tunable and narrow pore diameter [1], which made them ideal for use in energy conversion and storage devices. In particular, ordered mesoporous carbons are promising as supporting matrix for catalysts in fuel cells, hydrogen storage materials, electrochemical double-layer capacitors, and electrode materials in lithium batteries. The textural parameters of the nanoporous carbon materials are critical in many industrial applications particularly, separation, adsorption and fuel cells.

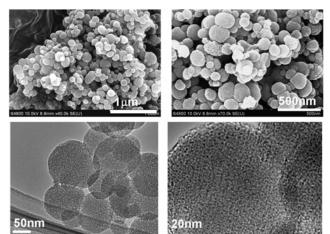


Fig: HRSEM and TEM images of mesoporous carbons

However, a little attention has been given in the studies to the textural parameters control of nanoporous carbon materials. The present study is focused on the preparation of ordered nanoporous carbon using mesoporous silica or metallosilicates as template and sucrose or glucose as a carbon source.

The carbon materials possess well ordered structure with regular particle size (Fig.). The specific pore volume is in the range of 0.93-1.51 cm³/g and pore diameter in the range of 1.6-2.3 nm. The specific surface area of mesoporous carbons prepared using glucose as a carbon source are in the range of 1384 m²/g to 2073 m²/g, which is higher than nanoporous carbons prepared using sucrose as a carbon source. The results indicate that the carbon source with a small molecular size is critical to achieve the nanoporous carbon materials with excellent textural characteristics. The textural parameters of nanoporous carbon samples can be finely controlled using glucose as a carbon source. In addition, the electro catalytic activity of the carbon materials has been studied. It has been found that the charge of hydrogen desorption is dependent on weight% of Pt loaded and textural parameters of mesoporous carbon materials. It has been found that mesoporous carbon shows superior electrochemical performance compared with carbon black due to its large ordered pore channels which are continuous connected with the large quantity of small pores in the carbon pore walls.

Reference:

1. P. Srinivasu et al., Adv. Funct. Mater. 18 (2008) 640.



Oral Presentation 3-3

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Presentation Title:

Theoretical investigation of Au(111) stepped surfaces: from structure to function

Abstract:

Metal surfaces might have a remarkably different structure with respect to the simple bulk termination. Many of them might undergo reconstruction processes. For example, Au(111) exhibits the so-called "herringbone" reconstruction that has been experimentally observed and theoretically justified. It has recently been shown that interesting reconstruction patterns arise in gold vicinals [1], where the usual alternating disposition of *hcp* and *fcc* regions of the flat surface is lost in favour of a more complex picture: the reconstruction lines are V-shaped, with hcp regions shrinking close to the lower step edge and getting larger at the upper step rim. Such reconstruction patterns have been recently used for controlling the growth of molecular networks and metal assembly, partly exploiting the different selectivity of the *fcc* and *hcp* termination towards adsorbates [2,3].

We are currently investigating Au(111) and its vicinals by means of computational methods, such as classical molecular dynamics and density functional calculations. The aim is to study the structure of the surfaces at the atomistic level and to extract information on those electronic properties that are able to explain the different surface reactivity of the *hcp* and *fcc* termination.

Finally the simulations will be compared to the experimental works that are already available in literature or that are currently being done at Empa laboratories.

References:

[1] S. Rousset et al., J.Cond.Matter **15**,S3363 (2003)

[2] M. Treier et al., Surface Science 602, L84 (2008)

[3] V. Repain et al., Materials Science and Engineering B 96,178 (2002)

Oral Presentation 3-4

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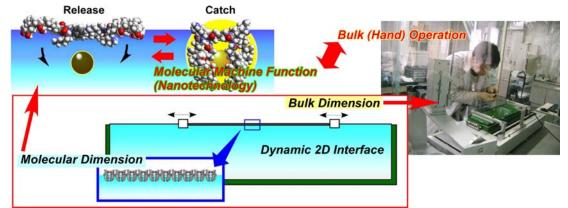
Home Page: www.nims.go.jp/super/HP/Ariga/A-top.htm

Presentation Title:

Supramolecular magic: hand-operating nanotechnology

Abstract:

Functional materials have been wisely constructed via bottom-up approaches as preparation of molecular patterns and complexes,¹⁻³ in organized seen nanostructures,⁴⁻¹⁰ and function materials.^{11,12} However, novel concepts to bridge nano (molecular) structures and bulk systems now becomes crucial in order to control real nano and molecular functions from our visible worlds. Here we propose a novel methodology "hand-operating nanotechnology" where molecular orientation, organization and even functions in nanometer-scale can be operated by our bulk (hand) operation. As shown in the following figure, this concept can be realized at dynamic two-dimensional medium, the air-water interface because this medium possess both features of bulk and molecular dimension. For example, we successfully manipulated molecules at the air-water interface upon bulk (10-100 cm size) motion of the entire monolayer and realized "capture and release" of aqueous guest molecules using molecular machine, steroid cyclophane.^{13,14} In addition, mechanically controlled chiral recognition by the armed cyclen monolayer was successfully demonstrated.¹⁵



References:

[1] Chem. Eur. J. 15, 2486 (2009). [2] J. Am. Chem. Soc., 131, 9494 (2009). Highlighted by Nature Chemistry. [3] Phys. Chem. Chem. Phys., 11, 6038 (2009). [4] J. Am. Chem. Soc. 130, 2376 (2008). [5] J. Am. Chem. Soc. 130, 4594 (2008). [6] Adv. Mater. 21, 989 (2009). Highlighted by Nature Asia Materials. [7] J. Am. Chem. Soc. 131, 4220 (2009). [8] J. Am. Chem. Soc., 131, 6372 (2009). [9] Adv. Funct. Mater. 19, 1792 (2009). [10] J. Am. Chem. Soc. 131, 11282 (2009). [11] J. Am. Chem. Soc. 129, 11022 (2007). [12] Angew. Chem. Int. Ed. 47, 7254 (2008). [13] J. Am. Chem. Soc. 122, 7835 (2000). [14] Langmuir 21, 976 (2005). [15] J. Am. Chem. Soc. 128, 14478 (2006).



Oral Presentation 3-5

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Presentation Title:

Surface and interface magnetism

Abstract:

Empa's function as a bridge between the fundamental and applied aspects of research received additional support in the area of magnetism since 2004 with the activities of the Nanoscale Materials Science Laboratory. Some examples of work in this area will highlight the potential of advanced magnetic force microscopy techniques for making quantitative analysis of magnetic thin film systems. Services to the industry- and research-examples will emphasize the applied side of our activities, for instance with the magnetic characterization of nanoparticles for biomedical applications. Technology transfer will be addressed with the example of a development in our laboratory of a commercial instrument which expands the range of conditions available to thin film characterization with scanning probe microscopy techniques.



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Presentation Title:

Internal and external nanostructuring of thin metal films

Abstract:

This paper focuses on novel ways of manipulating both internal and external geometries of thin metal films. The microstructure can be by tuned by high energy ion bombardment, which in contrast to current believe not only leads to grain growth, but also to selective grain growth (turning textured thin films into single - crystals) and grain rotation resulting in texture tilt. With regards to geometry changes the phenomena of controlled fracture for the fabrication of nanoplatelets, dewetting for the formation of nanoparticles and dealloying for the creation of nanoporous metals are addressed.

Oral Presentation 4-2

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Presentation Title:

New applications of the acoustic emission to monitoring of industrial installations

Abstract:

The Acoustic Emission (AE) measurement is potentially a very important technique, from the group of non-destructive testing methods, for the future. The capability of the computer processing providing the opportunity for monitoring the industrial installations in the operating conditions. The AE testing covers a wide spectrum, from monitoring the large pressure vessels, pipelines, storage tanks, and concrete structures. In case of the AE testing during operating conditions, when is no opportunity to increase the stress in the material, it is possible to execute the long-term or periodic monitoring. The AE monitoring is possible due to changes of flows, pressure, level of liquid and many others. The changes of the conditions during the operating in many cases allow to activate the potential source of AE. The application of AE to monitoring of industrial installations makes the possibility of preliminary evaluation of material technical condition and periodic verification of localized defect during normal operating conditions.

Oral Presentation 4-3

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Presentation Title:

Sacrificial passivation for microelectronics assembly

- engineering temporary diffusion barrier coatings

Abstract:

The aim of this work is to develop a joining technique with a nano-scale filler for high-power electronics packaging. Such joining process would utilize number of beneficial phenomena occurring at the nano-level, such as lowering the melting point, or shortening the diffusion distance. Therefore, it would enable a low-temperature, short-duration bonding method for the fabrication of high-temperature, highperformance joints. These efforts are motivated by the joining requirements and operational demands of wide band-gap semiconductor devices.

Engineering an intelligent thermally-temporary diffusion barrier coating, so called Sacrificial Passivation would preclude solid-state interdiffusion at the interface, yet allow the subsequent liquid phase reaction to proceed, once reaching the melting point of the interlayer. A benefit of precluding this reaction is the reduction in the requisite interlayer thickness even to the nano-scale. Using of the alloy constituents as nano-scale powders and nano-multilayers profits from reduction in the relevant diffusional length scale, and thus, the time required for homogenization.

The Sacrificial Passivation candidates have been selected and characterized for the use in Cu-Sn system, with Sn being the interlayer. The fundamental objective of this work is to identify both the intrinsic (material) and extrinsic (form and structure) properties of the coatings, which influence its behavior as a passivating layer for TLP system. The coating agents have been selected according to the decomposition temperature, degradation or decomposition products, as well as their coatability of the metallic surface. The samples were heated under protective atmosphere in the Rapid Thermal Annealing furnace to observe the stages of the microstructural evolution during heating. The solid state reaction and therefore a primary effectiveness of passivation have been evaluated by means of Differential Scanning Calorimetry.

- [1] N.S. Bosco, F.W. Zok; Acta Materialia, 2004
- [2] N.S. Bosco, B. Manhat, J. Janczak-Rusch; Scripta Materialia, 2008
- [3] K. K. Sosnowska, J. Janczak-Rusch, R. Spolenak; Proceedings Empa PhD Symposium, 2008
- [4] K.K. Sosnowska, J. Janczak-Rusch, M. Roth; Proceedings International Turbine Forum, 2009
- [5] K.K.Sosnowska, B. Manhat, J.Janczak-Rusch, N.S. Bosco; to be submitted



Oral Presentation 4-4

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Presentation Title:

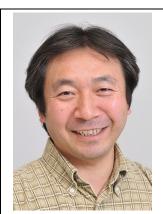
Development of new metallic membranes for hydrogen separation with a high hydrogen flux and the perfect selectivity

Abstract:

The production of high purity hydrogen is an important technical issue, which is related to future energy and environmental problems. Membranes of palladium alloys have been used commercially for decades to provide high purity hydrogen mainly for laboratory use and semiconductor manufacture. However, palladium alloys are too costly for large-scale production of high purity hydrogen. Keeping such situations in mind, we have developed some vanadium-based alloys for membrane materials to purify hydrogen gas to replace palladium[1-2]. Our vanadium-based membranes have acheived a high hydrogen permeation flux and the absolute separation coefficient, at the same time. Analysis of permeated gas using H_2 + He gas mixture revealed no sign of He gas pearmeation or leakage, which cannot be obtaind by nano porous membranes because the kinetic diameter of H_2 and helium is very similar. The vanadium alloys need thin palladium or palladium alloy films on surfaces to

The vanadium alloys need thin palladium or palladium alloy films on surfaces to protect from oxidation and to give catalytic activity to decompose hydrogen gas molecules into atoms to permeate the alloys. So, our membranes have a sandwich-type structure, which can be sometimes called metal-metal composite membranes. The membranes contain interfaces between bulk alloys and surface films. For the use at high temperatures such as 823 K, which is a typical operation temperature of membrane reactors for methane reforming, interdiffusion of bulk alloys and surface films can be a big problem. It is a common challenge for non-palladium based membranes to be overcome for hydrogen purification/ separation. In this study, the effects of crystal grain orientation of vanadium alloy substrate were investigated on the interdiffusion of vanadium and palladium. The long term permeation tests at 673 K clearly revealed that crystal grain orientations strongly affected the interdiffusion of vanadium substrate and paladium overlayer, suggesting a possible mitigation of interdiffusion at higher temperatures.

- [1] C. Nishimura, M. Komaki, S. Hwang and M. Amano, J. Alloys Comp. <u>330-332</u>, 902 (2002).
- [2] J. Y. Yang, M. Komaki and C. Nishimura, Int. J. Hydrogen Energy <u>32</u>, 1820 (2007).



Oral Presentation 5-1

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Presentation Title:

Biomaterials for bone tissue engineering

Abstract:

One of the main challenges in tissue engineering is development of suitable scaffolds. The scaffolds should mimic properties of natural tissue while providing a temporary matrix for growth of cells and formation of the new tissue. In particular, they should be characterized by high mechanical strength and 3D open porosity providing the space and transportation avenues for new tissue growth and then decay with the kinetics adjusted to new tissue formation and remodeling [1]. We hypothesized that development of the optimal construct with suitable stiffness and strength at each stage of new tissue formation requires use of different biomaterials arranged in the volume of the porous scaffolds in non-homogeneous and specific way.

The aim of the study was to develop a novel scaffolds, with honeycomb-like pattern, fully interconnected channel network, and controllable porosity, which can be fabricated in the form of layers of directionally aligned microfibres deposited via computer-controlled extrusion process. This required development of a novel method for optimization of the mechanical strength of biodegradable scaffolds against their degradation rate in the case of complex, non-homogeneous scaffold architectures.

The results showed that, application of different biomaterials for scaffold fibres is an efficient method of tuning up the mechanical properties and degradation rate of the construct with a significant influence on new tissue formations. In addition to that, the mechanical performance of the scaffolds may be also controlled by the lay-down pattern of the fibres. The fluid flow analysis showed that continuous and non-homogenous flow of the fluid through the designed scaffold can stimulate cell seeding and tissue differentiation. The preliminary in vivo and in vitro study confirm that the proposed 3D scaffolds fabricated using rapid prototyping method may be promising bone tissue engineering product.

Reference:

[1] Swieszkowski W. et al. Repair and regeneration of osteochondral defects in the articular joints. Biomolecular Engineering, 2007, Nov;24(5):489-95.



Oral Presentation 5-2

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Home Page:

Presentation Title:

Development of stepwise biomimetic matrices for regulation of stem cell functions

Abstract:

Cellular microenvironment including extracellular matrices (ECM) is an important factor to regulate stem cell differentiation. During tissue development, ECM are remodelled dynamically to regulate stem cell differentiation in vivo. Here, we developed a novel kind of "stepwise osteogenesis-mimicking matrices" that were supposed to mimic the in vivo developmental ECM by decellularizing serially differentiated mesenchymal stem cells (MSCs). The MSCs were cultured in proliferation or osteogenic induction medium to control their osteogenic differentiation at different levels. The ECM derived from non-differentiated, and differentiated MSCs at early and late stages were referred as stem cell matrices, early stage matrices and late stage matrices, respectively. The stepwise osteogenesis-mimicked matrices were used for culture of MSCs to investigate their effects on osteogenic differentiation. Alkaline phosphatase staining and gene expression analysis revealed that the early stage matrix enhanced osteogenesis of MSCs. The result could be explained by expression of osteogenesis-related transcription factors. MSCs cultured on the early stage matrix and late stage matrix expressed a higher level of RUNX2 than did those on stem cell matrix, suggesting that the stem cell matrix might directly inhibit the osteogenesis of MSCs. MSCs cultured on the late stage matrix expressed a high level of PPARG, transcription factor for adipogenesis, suggesting that the late stage matrix might suppress osteogenic differentiation by unexpected adipogenic differentiation. In conclusion, the stepwise osteogenesis-mimicked matrices could regulate osteogenic differentiation of MSC. They will provide a new model for the exploration of ECM on osteogenesis and be useful for tissue engineering.

Reference:

T. Hoshiba, N. Kawazoe, T. Tateishi, and G. Chen. Development of stepwise osteogenesis-mimicking matrices for the regulation of mesenchymal stem cell functions. *J. Biol. Chem. In press*.

Oral Presentation 5-3

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Home Page:

Presentation Title:

Preparation of nanostructured autologous scaffolds for tissue engineering

Abstract:

Extracellular matrices (ECM) play a crucial role in controlling and regulating cell behavior and function. Therefore, ECM and the ECM macromolecules have been widely used as artificial implants and scaffolds for tissue engineering and regenerative medicine. However, development of autologous extracellular matrix scaffolds (auECM) has been a great challenge for *in vivo* implantation without eliciting adverse inflammatory and immune responses due to the restricted availability of autologous donor tissues. Here we report a novel method to prepare auECM scaffolds by three-dimensional culture of cells in a removable porous skeleton. The auECM was deposited by cultured autologous cells in a three-dimensional porous template that could be selectively removed after ECM formation. The ECM scaffolds exhibited a porous nano-fibrous structure and showed excellent biocompatibility when being implanted. The auECM scaffolds are supposed to be useful in tissue engineering.

- 1. Griffith, L.G. & Naughton, G. Tissue engineering-current challenges and expanding opportunities. *Science* 295, 1009-1014 (2002).
- 2. Badylak, S. F. The extracellular matrix as a biologic scaffold material. *Biomaterials* 28, 3587-3593 (2007).

Poster Presentation 1

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Presentation Title:

Composites PLLA/MWNTs produced by twin-screw microcompounder

Abstract:

Carbon nanotubes (CNTs) have been attracting much attention due to their high electrical, thermal and mechanical properties. The properties combined with a high aspect ration made CNTs promising reinforcement of polymeric composites of high electric conductivity.

Possible advantages of modification with CNTs strongly depend on the their dispersion in the matrix. Proper dispersion of CNTs in the composite matrix is essential for efficient mechanical load transfer and for high electrical conductivity.

Several methods have been used to achieve efficient dispersion: stimulation with ultrasounds, melt blending at high temperature and shear forces; functionalization of CNTs; application of surfactants; in situ polymerization.

CNTs offer especially interesting possibilities when used in combination with biodegradable and biocompatible polymers. This is due to a large potential for biomedical applications, especially in bone tissue engineering. It has been also shown that CNT-reinforced biocompatible polymer composite can stimulate cell growth and regeneration by transferring the electrical signals to the osteoblasts As a result, electrical conductive scaffolds, CNTs/biocompatible polymer, in bone tissue engineering might be a good solution to improve bone regeneration and physical properties of the scaffold.

The objective of the study reported here is to develop a method to fabricate PLLA/MWNTs nano-composites in a controllable process without toxic products involved in other methods. Composites composed of the FDA-approved biocompatible poly(L-lactide) (PLLA) and multi-walled carbon nanotubes (MWCNTs) were fabricated by a melt blending process, using a DSM Xplore micro-compounder. This apparatus is a micro-scale twin-screw compounder, mimicking the mixing behavior of the large twin-screw extruders. The melting and blending in the compounder was tested for 2 different mixing times. MWCNTs content was 3 wt%. The microstructure of the obtained materials was examined by High Resolution Scanning Electron Microscopy. The size of nanofillers was analized with TEM. The

mechanical (tensile strength, Young modulus) and thermal (DSC, TGA) properties of nanocomposites were also investigated. The mechanical and thermal properties of the specimens have been correlated to their microstructure.

