THE SCIENCE AHEAD: U.S. SCIENCE IN THE 21st CENTURY

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The 20th century was so rich in scientific discovery and technological advance that it does not seem plausible for the 21st century to compare. The 20th century started with profound and revolutionary advances in our understanding of nature. As we begin the 21st century, there are significant opportunities for discovery in many areas of science, and in particular for the physical sciences. For example, in particle physics it can be argued that the situation at the dawn of the 21st century is in many ways similar to that at the beginning of the last century. Clues are emerging of radical changes to our understanding of the universe, and one can see that there are discoveries that will have a disproportionate impact on our understanding of nature and our universe.

In this talk, I will highlight some of the areas of physical science research that appear to be have great opportunity for discovery, some of the areas ripe for significant technological advance, and overview U.S. plans to address them.

THE GROWTH AND CHARACTERIZATION OF PHOTONIC THIN FILMS

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Photonic thin films have been grown on a variety of substrates using plasma enhanced chemical vapor deposition (PECVD) of organic monomers. Films produced by both mono-polymerization and co-polymerization have been prepared and analyzed. The films have been characterized by x-ray photoelectron spectroscopy (XPS), Fourier transform infrared spectroscopy (FTIR) and variable-angle spectroscopic ellipsometry (VASE). Studies of the mechanisms of the homo- and copolymerization reactions have aided in the optimization of the fabrication of photonic films. The effects of monomer feed-in rates were applied to manipulate the composition and chemistry of the deposited films to achieve the required optical properties. This presentation will emphasize the characterization of the polymerized films using XPS, and show how these results complement those using FTIR and VASE.
SELF ORGANIZATION PHENOMENA FROM KILO- TO NANO-METER SCALE: FROM DESERT DUNES TO WIRES AND QUANTUM DOTS

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Nature abounds in spectral colors and intricate shapes: the undulating ripples of a desert dune, the rainbow mosaic of a butterfly’s wing, the delicate curlicue of a grape tendril. All these creations delight the imagination and challenge our understanding. How do these patterns develop? What sorts of rules and guidelines shape the patterns in the world around us? Similar phenomena of "self organization" occur at nanoscale when low energy ion sputtering produces arrays of "quantum dots" or "quantum wires" on surfaces. These shapes can be easy controlled by changing a few macroscopic parameters like dose, energy, incident angle and flux of the ion beam or surface temperature [1]. Typically, this is a non-serial process that allows to pattern large areas with high short-range and some long-range order offering an interesting improvement at the current small scale production technologies.


ANTIFERROMAGNETISM IN SIMPLE OXIDE FILMS

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Antiferromagnets (AFM), unlike ferromagnets (FM), do not exhibit net magnetization and thus can not be controlled by or coupled to an external magnetic field. This basic fact made FM films the focus of many successful studies in the last decade, which in turn produced recent advances in the information storage technology. AFM are, on the other hand, far more interesting magnetic systems due to complex interplay between magnetic and structural order. It is thus expected that the AFM structures in lower dimensions will provide many new challenges and novel properties that are not found in FM films. The studies of AFM in thin films have only recently become possible by invention of novel techniques utilizing synchrotron radiation and spin-polarized tunneling. In this talk we will concentrate on magnetic properties of epitaxial NiO films as a prototype of a simple oxide with antiferromagnetic order. The topic covered will include (i) control the magnetic anisotropy of NiO films, both out-of-plane and in-the-plane using strain and stepped surfaces, (ii) attempts to control the AFM domain patterns in NiO films, and (iii) exchange coupling of AFM films to FM substrates and overlayers. The last topic is highly relevant to one of the most technologically important phenomenon exhibited by the AFM films know as exchange biasing, which will be also discussed.
Simultaneous ion bombardment during atom deposition changes the whole process of film nucleation and growth of a coating. The additional energy provided by the ions is shown to influence not only the nucleation and growth of deposited material, but also, to cause changes in film density, stress, phase, defect structure and microstructure.

For deposition of multi-component materials, the ion-assistance can also provide the fine control of added energy which causes one component in the system to segregate and form a nanocomposite structure. Metal nitride/silicon nitride systems are described in which grain boundary sliding between the components is reduced. These materials appear to have hardness well in excess of 40GPa and are referred to as ultra-hard coatings. Some recent data, obtained when using a dual ion beam system, shows the effects of temperature and added energy on the hardness of Ti-Si-N coatings. The films are seen to be stable even after baking to 1000 °C and retain their hardness and microstructure. Examples of these nanocomposite and other hard coatings already used in industry will be given.

The current methods for producing these coatings are described and include unbalanced magnetron sputtering and dual ion beam techniques. New studies with a lower energy ion beam system are needed to complete the parameter study. Whilst there is evidence that the ion bombardment increases the movement of atoms and the formation of nanostructures the damage from ions at energy above 100eV is already enough to break up this nanostructure.

Another application of energy-assisted coating is to produce thin layers of non-evaporable getter materials. Planar flanges have been coated by Ti-V-Zr and shown to pump CO when activated at 300 °C. A cylindrical magnetron system has been developed to allow coating of the inside walls of vacuum tubes with this Ti-Zr-V alloy.

Surface coatings also have a significant effect on the adhesion and proliferation of micro-organisms. Our results show that it is not only the surface chemistry which influences the cell adhesion, but also, the actual surface topography. Bacteria of different shapes and sizes are seen to prefer regular features of specific size and shape.
SELF-ORGANIZATION OF NANOSTRUCTURES IN SEMICONDUCTOR HETEROEPITAXY

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Exploitation of strain-induced self-organization phenomena during semiconductor heteroepitaxy is an elegant and efficient route towards fabrication of large-scale arrays of uniform nanostructures. This will be demonstrated for the Stranski-Krastanov growth of SiGe films on vicinal Si(001) substrates. In this system, the growth front undergoes a series of strain-relief mechanisms that include the formation of \{105\}-faceted three-dimensional crystallites and the evolution of a dislocation network. By tuning substrate miscut and film thickness or growing superlattices a variety of patterns with different symmetries can be obtained. Atomic-force microscopy has been applied to obtain quantitative information on nanostructure uniformity and arrangement as a function of growth conditions. The mechanisms of pattern formation will be explained in the framework of continuum elasticity theory. Analogies to pattern formation in heteroepitaxial growth of organic semiconductor films like para-hexaphenyl on a variety of substrates will be discussed.

Since these nanostructure arrays cover the entire wafer on which the films were grown, they can serve as large-area nanopatterned templates. This application will be demonstrated for the fabrication of nanomagnet arrays fabricated by shadow deposition of cobalt onto specially faceted SiGe films. The resulting nanomagnets are single-domain magnets as has been proven by magneto-optic Kerr effect (MOKE) and x-ray circular dichroism photoemission electron microscopy (XMCD-PEEM).

ADSORPTION DYNAMICS AND REACTIVITY OF RH/V ALLOYS

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Bimetallic surfaces have been used in heterogeneous reactions for a long time. The influence of alloy formation is explained in terms of both ligand effects and ensemble effects, which are very difficult to distinguish from each other. Recently two alloy systems (Pd – V and Rh – V) have been found where it may be possible to distinguish between ligand and ensemble effects. Detailed studies of the geometry and electronic structures of a Rh(111) – V subsurface alloy yield the necessary input to understand the reactivity of the alloy surface with respect to ligand and ensemble effects. Molecular beam studies show that the changes in the electrical structure due to alloying lead to significant alterations in the reactivity of the Rh – V subsurface alloy as compared to the V free Rh(111) surface. Experimental results obtained during studies with simple molecules (H\(_2\), CO and CH\(_3\)OH) are explained with respect to the differences in the electronic structures between Rh(111) and the Rh – V subsurface alloy. A change in the adsorption mechanism of dissociative hydrogen adsorption from dynamical steering to precursor mediated adsorption has been found. CO adsorption dynamics show that the extrinsic precursor is only stable on ordered CO adlayers, which are absent on the alloy surface. The reactivity of the pure and the alloyed surfaces towards the dehydrogenation reaction of methanol.
shows marked differences. The subsurface alloy has a significantly higher reactivity for methanol dehydrogenation than the V free Rh(111) surface. The latter result is important for the development of methanol powered fuel cells.

DIFFUSION IN NANOMETER-SCALE

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Diffusion on nanoscale is a very interesting, new field. For example we have shown recently [1-8] that, if the diffusion distance is comparable to the atomic spacing, the classical Fick I equation is valid only for composition independent diffusion coefficients, D. For strong composition dependent D (which is generally the case) surprising results are expected: sharp interface remains sharp and shifts linearly with time even in ideal binary solid solutions (like Cu-Ni). Furthermore, an initially diffuse interface can become sharp, which offers a very useful tool improving the quality of bi or multilayers produced for different applications (X-ray or neutron mirrors, semiconductor electronic devices based on Si/Ge multilayers). In general it is expected that deviations from the classical parabolic law (e.g. in solid state reactions) can be observed on nanoscale even if there is no chemical reaction control at all.

In the lecture results of computer simulations and measurements providing experimental evidences of the above phenomena in different bi- or multilayer systems (Si/Ge, Cu/Ni, Mo/V) will be presented.


NEW DEVELOPMENTS IN THE FIELD OF TOF MASS SPECTROMETRY WITH CONTINUOUSLY OPERATING ION SOURCE

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The operating principle of time of flight (TOF) mass spectrometer is simple. Ions are accelerated by a definite potential and left to run freely in a force free drift region. Having the same kinetic
energy, the smaller the mass, the faster the ion. The first ion to hit the detector will be the lightest, and the order of the arrival will be the monotonic function of the mass number.

A new concept for TOF mass spectrometry is presented. Currently, TOF instruments use pulsed ion introduction with a ten or so nanosecond pulse width, followed by a waiting period roughly 100 microseconds. Accordingly, the sample is under excitation in $10^{-4}$ part of the total measuring time, which limits the sensitivity of the method. Secondly, the ion bunch generated is strongly confined, which gives rise to space charge problems deteriorating the intensity and the mass resolution.

The concept presented uses a continuously operating ion source and a special dual modulation technique [1]. The expected advantages are higher sensitivity and minor space charge limitations.

There are two implementations of the new concept.

In **implementation "A"** two ion beam modulators are used. The first one at the entrance, the second one at the exit of the drift region right before the Secondary Electron Multiplier (SEM). The modulators are driven with the same sinusoidal driving voltage with increasing frequency in uniform frequency increments. The necessary detection bandwidth of the SEM and the electrometer is as low as some hundred Hz only, which enables to operate the electrometer electronics with unusual high amplification ($10^{10}$ V/A). There is a shortcoming, however, that the ion beam modulation method is not perfectly linear, thus the higher harmonic peaks inevitably show up in the spectrum. This can be reduced by low level modulation, but there is no way to eradicate it.

In **implementation "B"** there is just one ion beam modulator at the entrance of the drift region. This might still be nonlinear. The second modulation is carried out with an analog multiplier electronics right after the SEM and the electrometer. In here the SEM and the electrometer must have a high enough bandwidth (10 MHz or so) in order to deal with the high frequency modulated ion signal. The advantage in version "B" is, that the appearance of the higher harmonic peaks in the mass spectrum can be avoided, due to the almost perfect sinusoidal modulation of the analog multiplier electronic unit. Mathematical arguments will show, that at least one of the modulations must be perfectly sinusoidal in order to avoid higher harmonic peaks in the mass spectrum.

In both cases, the mass spectrum will be the result of the Fast Fourier Transformation (FFT) on the output signal.

Because of the high signal level, it was possible to omit any electrostatic mirror configuration for bunching the ions. Rather a cylindrical energy filter was installed to remove the ions with undesired energies. The drift tube is roughly two meters long.

Lead on silicon represents a prototype system of metal/semiconductor interface since Pb and Si do not form any compound. The system shows a variety of structures at Pb coverage lower than 1 ML on the Si(111)/(7x7) surface, forming 2D alloys with silicon atoms. The interface is abrupt, making the interpretation of results more straightforward. At a coverage of 1/6 ML, the mosaic-\(\sqrt{3}x\sqrt{3}\) structure forms and consists of alternating rows of Pb and Si atoms. This structure exhibits quantised valence states. At slightly higher coverage of Pb surface the commensurate \((\sqrt{3}x\sqrt{3})R30\) phase co-exists with the incommensurate 1x1 islands. The interface between these two phases fluctuates substantially at temperatures \(\geq RT\). Particular phases and corresponding phase transitions were studied using SRPES and VT STM to understand their stability and the driving force behind the transitions. As a main experimental tool the doping of layers with Cs and O atoms was used to identify the charge transfer between Si and Pb and particular states of these atoms.

During the last years, increasing attention has been paid to the interface properties of multilayered structures. It was shown that both the interface roughness resulting from the growth process itself and the interface mixing of constituents affect the quality of a multilayer stack and its applications as an interference mirror for X-ray optics. In our works the preparation (mostly by electron beam but also by sputtering and MO CVD) of mirrors for standard applications around the wavelength of 13.5 nm and their thermal stability under different types of annealing was studied. The basic bilayers or tetralayers of mirrors were W/Si, Mo/Si, \(W_{1-x}Si_x/Si\), Co/Si/W/Si. In the paper the interface properties of structures studied by X-ray reflectivity, diffuse scattering and cross-sectional TEM vs. their stability are summarized. The detailed knowledge of the interfaces becomes crucial for nanometer-scale period multilayers with layer thickness below 1 nm. They determine the progress of the soft X-ray examination of biological objects and they are promising for hard X-ray optics. Here Sc/Cr multilayers are reported. Sc/Cr based mirror is one of the best candidates for "water window" range of wavelengths. Attention is paid to the characterization of interfaces of the stack with 250 periods, bilayer thickness of 1.75 nm and interface roughness controlled at the 0.2 – 0.3 nm level.
NANOSTRUCTURE AS SEEN BY THE SAXS

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Small Angle X-ray scattering (SAXS) has been successfully applied on size and shape investigation of nanometer size features in different fields. With the rise of interest in nanoparticles in material science, the method is fully employed in research of wide variety of materials. Finally, the advance of thin films research with the structural features in nanometer range also shows great need for SAXS, albeit in the grazing incidence set up (GISAXS).

The sensitivity of GISAXS on the surface roughness is employed in the investigation of the quality of the film growth while the thickness and the density of the film are determined precisely. The size and shape of the nanostructures, be it particles or vacancies, precipitations or agglomerations, that are present in the film are easily obtained, as well as the information about their depth distribution. The presence of nanoparticles on the film surface, or on the interface between different films in form of islands is clearly resolved.

A good illustration is the SAXS study of magnetron sputtered tungsten carbide thin films. The surface roughness varies with the carbon concentration, while the films are built solely of nanosized tungsten carbide grains and of these grains surrounded by amorphous carbon matrix, for low and high carbon concentration, respectively. The size and the distribution of these grains has been determined with the combination of both transmission and grazing incidence SAXS.

QUANTUM OPTICS OF DISPERSIVE DIELECTRIC MEDIA

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We quantize the electromagnetic field in a polar medium starting with the fundamental equations of motion. In our model the medium is described by a Lorenz-type dielectric function \( \varepsilon (r, \omega) \), appropriate e.g. for ionic crystals, metals and inert dielectrics. There are no restrictions on the spatial behavior of the dielectric function, i.e. there can be many different polar media with arbitrary shapes. We assume no losses in our system so the dielectric function for the whole space is assumed as real. The quantization procedure is based on an expansion of the total field (transverse and longitudinal) in terms of the coupled (polariton) eigenmodes, and this approach incorporates all previous results derived for similar but restricted systems (e.g. without spatial or frequency dependence of coupled modes). Within the same model, we also quantize the Hamiltonian of a nonretarded electromagnetic field in polar media. Particular attention is paid to the derivation of the orthogonality and closure relations, which are used in a discussion of the fundamental (equal-time) commutation relations between the conjugate field operators.

As an example, we shall apply our theory to the quasi-two-dimensional Wigner crystal, formed by electrons at very low temperature. In particular, we shall discuss the influence of the quantized electromagnetic field on the dynamics of Wigner electrons, i.e., on the dispersion relation of Wigner phonons.
INFLUENCE OF DEEP-CRYOGENIC TREATMENT ON WEAR RESISTANCE OF VACUUM HEAT-TREATED HSS

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In this paper we compare the wear behaviour of a vacuum-heat-treated ESRAISIM2 high-speed steel and the same steel that was vacuum heat treated in conjunction with a deep-cryogenic treatment at –196 °C. Four different tempering temperatures for the specimens austenitized at the same austenitizing temperature were carefully selected to obtain various in-advance-determined combinations of the Rockwell-C hardness and the fracture toughness $K_{IC}$. Each of the eight specimens was therefore characterised by these two resulting material properties. The wear behaviours were then compared and discussed in terms of these two properties and the related microstructures. The wear study was performed using a reciprocating sliding device under well-controlled contact conditions. Relatively high loads were used to provide enough wear for a comparison of the selected samples. A much harder and dissimilar model counter-material, i.e. silicon nitride ceramic, was used in order to avoid excessive wear of the counter samples and adhesion, which could occur in contacts with similar materials (metals/steels) under such high loads and in non-lubricated conditions. The wear behaviours were then compared and discussed in terms of these microstructures and the related properties. The differences in the wear resistance obtained in our investigation were as high as an order of magnitude. However, the beneficial effects are not a direct result of the type of the treatment, but relate to a proper combination of the resulting fracture toughness and the hardness. The more uniform and moderate values, which, however, tend to be obtained with a deep-cryogenic treatment, are beneficial to the high wear resistance of the selected high-speed steel.

STABLE OPERATION OF FIELD EMITTERS – AN OPEN CHALLENGE FOR THE VACUUM SCIENCE

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Field emission phenomena attracts theoretical physicists for several decades and the basic mechanism is well understood today. The technology of manufacturing various materials in the form of sharp tips improved greatly in the meantime as well. Namely, a driving force for scientific and technological activities during all decades was the idea to replace power consuming thermionic cathodes by field emitters in various e-beam devices. The great shortcut towards simple preparation of sharp tips was the discovery of nanotubes, recognized soon afterwards to be field emitters with attractive properties. Several scientific and technical papers manifest their advantage in prototype x-ray tubes, flat panel displays, microwave tubes, etc. Unfortunately, great expectations of potential customers were hindered by a simple fact that the degradation of their performance was too fast.

The analysis of a field emitter degradation is thus closely related to general problems studied in vacuum science and technology. The crucial ones are: is it possible to measure the intrinsic...
performance of a field emitter, what is the influence of the residual atmosphere, can the
degradation be predicted from an accelerated test in a specified gas, what determines the residual
atmosphere in a sealed device? In the contribution, an overview of present-day instrumentation
techniques, which can help to answer the above set questions, is made and supported by some
latest and original results.
PREPARATION AND CHARACTERISATION OF ADSORBED C\textsubscript{2}H\textsubscript{5}Zn ON Pd(100) AND Rh(111) SURFACES

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Dialkyl zinc compounds are widely used as reagents in organometallic chemistry. Of particular importance is their role as alkylation and arylating reagents although the Grignard reagents are the best known and widely used ones. The high reactivity of dimethyl and diethyl zinc (DEZn) stem largely from the polar nature of the zinc-carbon bond. The alkyl zinc compounds can also be used as additives in polymerisation or as a zinc source in wide-gap II-VI semiconductor technology, such as ZnSe, as well as in the application of solar cells. The growing interest in bimetallic systems and the catalytic importance of Pd/ZnO in methanol synthesis are of practical importance.

The adsorption of DEZn on Pd(100) and Rh(111) surfaces has been studied in detail [1,2]. The formation of monoethyl zinc (MEZn) has been confirmed by methods including photoelectron (UPS, XPS), Auger-electron (AES), electron energy loss spectroscopy (EELS and HREELS), temperature programmed desorption (TPD) and work function measurements.

It was found that besides the main products – atomic Zn, and C\textsubscript{2}H\textsubscript{5} species – in the temperature range from 90-200 K C\textsubscript{2}H\textsubscript{5}Zn, MEZn, chemisorbed DEZn and condensed DEZn were also present on both surfaces. The relative amount of the components depends on the temperature and exposure. The photo-induced mechanism resulted in a simpler feature. By photons (hv~5.3 eV) the monolayer DEZn at 90 K was completely transformed into a layer of MEZn, Zn and C\textsubscript{2}H\textsubscript{5} species. MEZn is best characterised by its Zn 2p\textsubscript{3/2} XPS peak at 1022.5 eV and was found stable up to 240 K.

The goal of this work is to compare the chemical reactivity of adsorbed MEZn on Rh(111) and Pd(100) surfaces.

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CHARACTERIZATION OF Ge ISLANDS ON SI(100) SUBSTRATES

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The growth and the self-organization of Ge islands on Si substrates have been extensively studied in the last few years not only because of their great technological potential but also to understand the fundamental process of thin film growth.
We present a preliminary study of Ge islands formation on Si(100) substrates using grazing-incidence small-angle X-ray scattering (GISAXS). GISAXS is very powerful technique for the characterization of islands supported on a substrate. From the 2D GISAXS pattern it is possible to determine the shape, size and inter-island distance.

Samples in our study were prepared by a high-vacuum evaporation of a 5 nm thick Ge layer on Si(100) substrate held at 200 °C. The samples were subsequently annealed at 700 °C for 1h in vacuum, yielding to island formation. A Fortran program IsGISAXS was used for the simulation and analysis of Ge islands.

Vertical cut (perpendicular to the surface) of the experimental 2D GISAXS pattern has been fitted using a Guinier approximation. Obtained parameters were used for the simulations. The simulated 2D GISAXS pattern well reproduce experimental data for cylindrically shaped islands with morphological parameters R=4 nm, H/R=0.25 and the average inter-island distance D=5 nm.

**AB INITIO CALCULATIONS OF Xe MONOLAYERS ADSORBED ON Cu(111) SURFACES**

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The initial adsorption of xenon on Cu(111) surfaces occurs into hexagonal monolayers, which at temperatures around 77K form a ($\sqrt{3}$x$\sqrt{3}$)R30° commensurate structure. There has been considerable interest in the properties of this system, in particular concerning the contribution of chemisorptive bond to the overall adsorption energy. We have performed ab initio density functional calculations of the system and determined the equilibrium distances, force constants, and adsorption energies. We have found that DF calculations with the GGA functional, to which the Van der Waals interaction is explicitly added, give excellent agreement with experiment. The chemisorptive bonding is important in determining the equilibrium distances and force constants, but contributes comparatively little to the binding energy of both Xe-Xe and Xe-Cu bonds. The use of the asymptotic form of the Van der Waals interaction appears appropriate, but the position of the image plane of the metal must be carefully evaluated. We find a small preference for the on-top adsorption site, in agreement with experiment.

**STRUCTURAL STUDY OF EPITAXIAL TUNGSTEN OXIDE NANOCYLUSTERS**

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Tungsten oxide is widely used in a variety of electrochromic devices, catalysers and chemical sensors. One of the characteristic properties of tungsten oxide is that its electrical conductivity
depends strongly on the oxidation state, which can vary from the wide semiconductor state (for WO$_3$) to the conductor one (for WO$_2$). A number of other phases of WO$_x$ exist between these two principal states. They differ not only in their composition and oxidation state but also in the crystallographic structure. The structure and morphology have a large influence on the sensitivity, selectivity and stability of the gas sensors and catalysts.

The tungsten oxide nanoclusters were prepared by evaporation of tungsten oxide powder from a specially designed evaporation cell under UHV (ultrahigh vacuum) conditions. Several surface orientations of $\alpha$-Al$_2$O$_3$ single crystal were used as substrates. The structure of tungsten oxide nanoclusters was determined by RHEED (Reflection High-Energy Electron Diffraction). The crystallographic structure and epitaxial parameters of nanoclusters were found to be dependent on the substrate surface structure and deposition conditions. The well-oriented populations of WO$_2$ nanoclusters were observed. They exhibited preferentially (101) WO$_2$ epitaxial plane. The chemical composition as well as the oxidation state of tungsten oxide was determined by XPS (X-ray Photoelectron Spectroscopy) method.

DEVELOPMENT OF THE SILVER 4d BAND IN ULTRA THIN FILMS ON Cu(100), Ni(111) AND Pd(111) SURFACES

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Using a high resolution energy and angle resolved photoemission spectroscopy (ARPES) we have studied the development of the silver 4d band in ultra thin silver films deposited on Cu(100), Ni(111) and Pd(111) surfaces. The structure of the films has been investigated by low electron energy diffraction (LEED) and scanning tunnelling microscopy (STM). In all studied cases silver forms films of fcc structure though, due to different mismatches with the substrate surfaces, with different levels of distortion. The structure of the overlayer film and the interaction with the substrate were correlated with the structure of the silver 4d band. In the case of the copper (100) surface, the structural changes from the dispersed isolated silver atoms in the form of the surface alloy to the saturated monolayer was accompanied by the change of the spin orbit splitting. In contrast, the high resolution ARPES measurements do not indicate any change in the spin orbit splitting during the monolayer formation on Ni(111) and Pd(111). The transformation of the low dimensional Ag 4d band into the fully developed bulk band is compared with the Ag/V(100) system where d-states are quantized.

THE INFLUENCE OF CARBON ON THE QUATERPHENYL THIN FILM GROWTH ON A GOLD(111) SURFACE

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The tailoring of the growth mode of ultra-thin organic films is of utmost importance for the application of electronic and optoelectronic devices. As a model system we have studied the system p-quaterphenyl on Au(111), where we have focused on the influence of carbon pre-coverage on the structure and morphology of the thin films. Various analytical methods, like TDS, XPS, LEED, XRD and AFM have been applied for these investigations. On the clean Au(111) surface quaterphenyl forms a regular monolayer structure. The molecules are lying with their long axes parallel to the surface and are arranged with their short axes alternately parallel or side tilted. Furthermore, the molecules are azimuthally aligned along the dense packed rows of the (111) plane. The arrangement of the molecules in the multilayer (thin film) is similar to the monolayer structure, revealing an epitaxial growth. Regarding the morphology the multilayer consists of needle like islands. Small amounts of carbon (0.15 ML) change the monolayer structure (only side tilted molecules), as well as the multilayer structure and morphology. The azimuthal locking of the molecules in the multilayer is lifted and consequently the morphology is changed. For a 0.5 ML carbon pre-coverage (saturation coverage) the azimuthal locking of the quaterphenyl molecules is already lifted for the monolayer. In the multilayer the molecules are arranged with their long axes more or less normal to the surface (standing molecules). This in turn changes the morphology of the thin quaterphenyl film to a continuous layer, consisting of small platelets.

THE OXIDATION OF A Mo₂C/Mo(100) SURFACE:
XPS, AES, LEIS AND MS STUDY

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The interaction of oxygen with a molybdenum carbide surface was investigated at different temperatures (300 K-1100 K) by means of XPS, AES, LEIS (low energy ion scattering spectroscopy) and MS. The different information depths of XPS, AES and LEIS (with topmost layer sensitivity) allowed us to obtain a depth profile of the sample.

The Mo(100) surface was carbidized by repeating C₂H₂ adsorption at 900 K and annealing in vacuum to 1265 K until the C content reached saturation. According to angle-resolved XPS measurements a carbide overlayer with a constant Mo₂C stoichiometry down to the information depth of XPS was obtained in this manner.

The removal of C from the outermost layer by O₂ (in the form of CO) was observed at 5-600 K and also at low O₂ exposures at 800 K. It allows more O atoms to adsorb on free metallic sites of the first layer. Applying higher doses of O₂ at 800 K results in a reconstruction of the surface region accompanied by the inward diffusion of O and the partial oxidation of Mo atoms. The
removal of C from subsurface sites occurs, however, only from 900 K. At $T_{ads} = 1000$ K the amount of adsorbed oxygen is smaller, but the carbon content of the subsurface region is effectively diminished, too.

**ON THE CALIBRATION OF THE EPES SPECTROMETERS**

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Elastic Peak Electron Spectroscopy (EPES) belongs to the electron spectroscopic methods of surface analysis. It makes use of the energy (E), elemental (Z) and angular ($\Omega$) dependence of electron elastic reflection coefficient $\eta = \eta(E, Z, \Omega)$. Yet, EPES has been successfully used in monitoring of ultrathin films growth and it has attained a significant position among other techniques due to its unique application – experimental determination of the electron inelastic mean free path (IMFP).

In frame of this contribution we present a novel method for (absolute) calibration of the Retarding Field Analyser (RFA) type EPES spectrometers. A calibration method consisting in application of a negative target bias slightly exceeding the primary voltage and thus giving rise to a quasi-spectrum formed by the primary electrons repelled backwards to the analyser without touching the sample surface had been elaborated by Dolinski et al. Later it was sophisticated by Bideux et al. We developed it further and called it QEPES. Namely, it has been shown that under such circumstances the trajectories of electrons between the sample and the analyser are no more radial, which leads to significant distortion of the QEPES peaks. Therefore, a special procedure how to estimate the QEPES intensities in order to exclude the spectrometer response function and to normalise the EPES intensities was needed. We invented one and demonstrate its performance.

**ELECTRONIC STRUCTURE OF Be(0001) SURFACE AS SEEN BY ARPES WITH VARIABLE POLARIZATION SYNCHROTRON RADIATION**

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We measured the electronic structure of the Be(0001) surface using high-resolution angle resolved photoemission (ARPES) with variable polarization synchrotron radiation. We used photons of 32.5 eV and 86 eV delivered by the beamline APE at Elettra, with the two orthogonal linear polarizations. The electronic band structure along the two ($\Gamma K$ and $\Gamma M$) high symmetry directions was probed. As a function of photon polarization either states of even or odd symmetry with respect to the scattering plane (defined by the direction of light and the
propagation direction of the measured outgoing electrons) could contribute to the photoemission intensity.

We observe the surface states around Γ and M and the surface resonance around M (Bartynski et al., Phys. Rev. B 32, 1921 (1985)). For the Γ surface state we find that a significant contribution to the photoemission intensity is present also in the 'forbidden' geometry, i.e. with the vertical polarization, while in the case of M surface state the contributions of the even and odd states are almost the same.

We will discuss the photoemission intensity for Be(0001) by comparing data to the available theoretical results.
A strong satellite line was observed and interpreted as plasmon excitation in Ge KL$_{23}$ Auger spectra [1]. Plasmons can be created due to two excitation processes. *Extrinsic* plasmons are excited when an electron traveling in the medium perturbs the static electric field of the lattice and generates density oscillations in its free (or near free) electron sea. In the case of Auger- or photoelectron emission, however, the sudden appearance of a positive core-hole may cause similar oscillations, with the same quantised energy loss, called *intrinsic* plasmon excitation. In order to separate the loss contributions of the different plasmon excitation processes a spectral evaluation method, based on different inelastic background correction procedures was developed [1]. In this work the dependence of the extrinsic and intrinsic plasmon excitation probabilities on the thickness of the emitting layer is investigated. Ge overlayer samples with thicknesses between 15-100 nm on single crystal Si substrate were prepared using DC magnetron sputtering technique [2]. Ge KL$_{23}$ Auger spectra were measured using the home-built high energy resolution ESA-31 electron spectrometer [3]. In order to interpret our experimental results theoretical calculations based on the dielectric approach are planned to be employed.


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**SIMS STUDY OF Ti-Zr-V NEG THERMAL ACTIVATION PROCESS**

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In this work properties of non-evaporable getter (NEG) films prepared on stainless steel substrates by magnetron sputtering were investigated. Changes of the sample surface during thermal activation were studied by means of Secondary Ion Mass Spectroscopy. Static SIMS observation of superficial layer as well as dynamic profiling of the surface region was performed.

Two samples of the same Ti:Zr:V stoichiometry were investigated following two different procedures of thermal activation. Step by step heating up to 280 °C (as model activation) and direct heating to 240 °C were applied. The SSIMS measurements are highly surface sensitive and reflect the changes of the superficial oxide layer covering air exposed surfaces during activation. To compare final states of activation the depth profiles of surface layer have been used as well. Molecular ion intensity ratios MX' / M' (X = O, C, H, OH; M = Ti, Zr, V) have been
considered to be directly coverage sensitive and were monitored during processes. The results were checked by comparison to corresponding XPS experiment.

NEW REACTION ROUTE OF HCOOH CATALYTIC DECOMPOSITION

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The adsorption and surface reactions of formic acid on TiO₂ and TiO₂-supported metals are important in many respects. Formic acid can easily dissociate producing formate and hydrogen [1,2] on these surfaces. Formate decomposes further to a variety of products, the dominant of which are CO, CO₂, H₂O and H₂. Taking into account these products, the decomposition of formic acid on TiO₂ surfaces has been described in terms of dehydration (to CO and H₂O) and dehydrogenation (to CO₂ and H₂) mechanisms [3-8]. Recent studies on single crystal TiO₂ surfaces revealed that the production of H₂O and CO could not be directly linked, thus the unimolecular dehydration reaction can be excluded [9]. In the present study the adsorption of HCOOH and the further reaction of the produced adsorbed species on TiO₂ and on TiO₂-supported noble metals (Rh, Pt, Au) has been studied by FTIR and mass spectrometry. Experiments on TiO₂ (110) and Rh, Pt supported on TiO₂ (110) single crystal surface were performed with the help of RAIRS, AES and TPD methods, and the results obtained on polycrystalline and single crystal surfaces were compared. Besides molecularly adsorbed HCOOH and surface formate species, adsorbed formaldehyde was detected by FTIR. MS measurements showed formaldehyde in the gas phase, too. The production of formaldehyde (both in the adsorbed layer and in the gas phase) depended on the pretreatment, the metal content of the catalysts and on the reaction temperature. The formations of formaldehyde and CO were complementary, from which it was concluded that CO forms mainly in the decomposition of formaldehyde formed in HCOOH deoxygenation. Further studies of formaldehyde surface reactions strengthened the above statement.

Epitaxial thin films of gallium nitride prepared by metalorganic chemical vapor deposition (MOCVD) are very promising for practical applications. Therefore it is necessary to know their properties. In this contribution we will present the results of atomic force microscopy (AFM) analysis of the morphology of the upper boundaries of GaN films prepared on sapphire substrates. Moreover, we present results of AFM analysis of GaN nucleation layers that are formed in the first stage of GaN thin films deposition process. It will be shown that GaN films exhibit randomly rough upper boundaries. The values of the main statistical quantities describing this roughness determined, i.e. the values of the standard deviations of heights and slopes, one-dimensional distribution of the heights and power spectral density function, will be presented as the functions of the thickness of the GaN films. It will be also shown that the topography of nucleation layer is formed by isolated islands. The values of the main quantities of the statistical roughness and the values of the quantities describing the island structure, i.e. mean island size and island size distribution, will be determined for different technological conditions used for nucleation layer preparation.

Tin (Sn) deposited on polycrystalline Aluminium (Al) surfaces shows a Stranski-Krastanov growth mode which means that before Sn-islands nucleate a thin Sn-wetting layer is formed (Eisenmenger-Sittner, J. Cryst. Growth 205, 1999, 441-452). If the wetting layer is removed from the Al-surface by sputter cleaning in UHV it re-forms by a solid state wetting process (Eisenmenger-Sittner at al., Surf. Sci. 489, 2001, 161-168). The sources of the Sn-atoms forming the wetting layer are the Sn-islands which are not removed from the Al-surface due to their size. Nonetheless it was observed that not every island acts as Sn-emitter.
In this work we show that this selective Sn-emission is a consequence of the chemical composition and the crystallographic structure of the Sn islands. Regarding the chemical composition of the Sn islands each residual trace of Oxygen has to be removed to facilitate the emission of Sn-atoms from the island boundaries. In addition wetting is only initiated if the sputter cleaning process results in a visible damage of the islands thus increasing the roughness of their surface. From this rough interface Sn-atoms can emerge more easily than from a crystallographically smooth surface due to their lower coordination.

By monitoring line-scans obtained from Scanning Auger Electron Spectroscopy the diffusion coefficient of the Sn-atoms could be estimated from the progress of the rim of the wetting layer around the islands.

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MORPHOLOGICAL INVESTIGATIONS ON Cd₄GeSe₆ CRYSTALS AFTER ELECTROCHEMICAL LAYER REMOVAL

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Solar cell technology is a technology of microelectronics that has already demonstrated its effectiveness, and holds great promise in generating electrical energy for the word. One of the greatest problems in the solar cell application is the storage of electrical energy. This problem may be solved with the help of electrochemical solar cells, which are suitable to generate either electrical energy or hydrogen under the appropriate conditions. There are some technical and scientific problems in the realization of electrochemical solar cells. One of the greatest tasks is to solve photo corrosion which ruins the semiconductor electrode during the operation of solar cells. The possible direction of this research is to search for novel materials that have appropriate properties for electrochemical energy conversion.

Cd₄GeSe₆ will be presented in this work, which is a new material for purposes of electrochemical solar cells. We are investigating the properties of this material that have been scarcely investigated and not all of its properties are known in details. Cd₄GeSe₆ belongs to the agryrodite family of which lattice parameters were determined. The band gap and type of band transition was determined by absorption and photoelectrochemical method. Furthermore we found that this material shows very good resistivity against photocorrosion. In this work we also determined the electrical parameters of the Cd₄GeSe₆/electrolyte junction, which is a very important property in making solar cells.

After electrochemical etching the surface generally remains structured. The roughness of the etched surface depends on many factors. Under special etching conditions the morphology of the etched surface is related to the defect density in the crystal. Under certain etching conditions the surface morphology displays fractal behaviour. The surface morphology will be studied with the help of image processing and box counting method, where the pattern recognition is carried out with the grade of membership method.

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INTERACTION OF SLOW ELECTRONS WITH SOLIDS: ROLE OF UNEOCCUPIED ELECTRONIC STATES IN SEES FINE STRUCTURE FORMATION

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The fine structure of experimental true secondary-electron emission spectra (SEES) along the normal to a clean W(110) surface is interpreted theoretically. In addition, a comparison to existing theoretical and experimental data is given. It is shown that the fine structure of SEES essentially depends on the quality of target surface preparation in the ultrahigh vacuum and is mainly due to the electronic structure of unoccupied high-level electronic states (above the vacuum level $E_{\text{vac}}$), which become occupied by electrons entering the solid. The predominant role of the bulk energy-band structure $E(k)$ effects in the spectra formation is shown. The extrema in SEES reflect the energy position of the critical points in the unoccupied $E(k)$, such as band edges or extremal dispersion-branch curvature points. And there occurs a possibility for the experimental study of the electron dispersion in the region of energies much higher than $E_{\text{vac}}$ (thus adding to the traditionally used data of the photoemission, inverse photoemission and optical spectroscopy) and for usage of the SEES data in a more perfect $E(k)$ calculation showing which singularities of the spectra relate to some or other bands. The method being developed enables one to distinguish between the bulk effects in SEES from the surface ones, which are to be investigated separately.

REACTION AND DESORPTION KINETICS OF H₂ AND O₂ ON ACTIVATED AND NON-ACTIVATED PALLADIUM SURFACES

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Within this study the influence of different methods of hydrogen supply towards the palladium surface on the water formation has been investigated, as well as the change of the reaction and desorption characteristics induced by a modification of the surface. Hydrogen has been dosed either in molecular form, in form of gas-phase atomic H or by diffusion of hydrogen to the surface, using a Pd-permeation-source. Oxygen has always been dosed in molecular form. Modification of the surfaces (either a Pd(111) single crystal or a polycrystalline palladium sample) has been achieved by deposition of different amounts of potassium. Temperature programmed reaction spectroscopy on the water formation reaction in the UHV regime has been carried out. From these measurements temperature dependent sticking coefficients for H₂ and O₂ under reaction conditions could be obtained. Additionally, optimum reactant supply ratios and abstraction probabilities for atomic hydrogen have been determined. Time-of-flight spectroscopy allowed the measurement of the mean kinetic energy of the permeating and desorbing hydrogen, oxygen and the H₂O reaction product. Significant differences in the reaction and desorption characteristics for the clean and modified palladium surfaces could be demonstrated. In particular, the occurrence of a strong hyperthermal component in the
desorption flux of permeating hydrogen from the potassium modified Pd surfaces could be observed. This indicates the build-up of an activation barrier for desorption in the potential energy surface. The results of these investigations should lead to a better understanding of basic steps in catalytic reactions and may be helpful in the effort to tailor specific catalytic processes.

**BALLISTIC EFFECTS IN ION BEAM MIXING**

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Molecular dynamics simulations have been used to study the driving force of ion irradiation induced interfacial mixing in metal bilayers in which the relative mass of the constituents is considerable. We find no apparent effect of chemical forces, such as heat of mixing or cohesive energy up to 8 keV ion energy, although a considerable number of liquid and high energy particles (hot atoms) persist up to even ps during the thermal spike.

It is in direct conflict with the widely accepted theory of thermal spike mixing (chemical interdiffusion model). The supersaturation of vacancies also occurs and which induces a thermally activated intermixing of the lighter constituent of the bilayer. The delay and the decoupling of the intermixing of the light constituent is explained as a backscattering effect at the interface: the interface acts as a diffusional barrier for high energy particles due to the large difference in atomic masses. The heavier atoms are predominantly ejected to the overlayer at the beginning of the thermal spike while the light atoms are injected to the bulk at the beginning of the cooling period (in Ti/Pt) or during the thermal spike with some time delay (Al/Pt).

Further details are given in http://www.mfa.kfki.hu/~sule

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P. Süle, M. Menyhárd, K. Nordlund, Nucl Instr. and Meth. B, accepted for publication  
http://babbage.sissa.it/condmat/0310238

**THE ELECTRONIC STRUCTURE OF ULTRATHIN Ag FILMS ON Pd(111)**

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We have performed *ab initio* density functional calculations of 1 to 3 monolayer films of silver on a Pd(111) surface. Due to the similarity of the lattice constants, silver layers grow in register with the substrate. We have in particular studied the electronic structure, and the formation of quantum well (QW) states in the overlayer films. The s states of silver which are close to the Fermi level hybridize strongly with palladium states, while the d states at binding energies around 4 eV do not have corresponding palladium states, at least around the centre of the surface Brillouin zone, and can form QW states. This is in agreement with recent photoemission experiments on such systems. We discuss the predictions of our calculations for a range of film thicknesses and angles in angularly resolved photoemission.
Low activation temperature Ti-Zr-V non-evaporable getters (NEG) developed in CERN gradually find their place in a variety of R&D and industrial applications. Nevertheless, mechanisms involved in their thermal activation and pumping action are not fully explained yet. Understanding these processes is essential to further development of NEG coatings, particularly to lowering the activation temperature determining the scope of their practical use.

Relative simplicity of Zr-V binaries exhibiting similar properties as Ti-Zr-V NEG was found advantageous to get insight into activation and gettering mechanisms. Studies of Zr-V NEG show that metallic components (oxidized after air-venting) are reduced during the thermal activation, activated surface is zirconium enriched, residual suboxides remain in the top surface layers and carbides formed during activation are located in subsurface region.

In this work, surface characteristics of the activated Ti-Zr-V NEG have been studied by means of surface sensitive techniques (XPS, LEIS, SIMS). As expected, many similarities with binaries have been found — top surface layer is zirconium enriched and residual suboxides are observed on activated surface. In contrast to the binary NEG, the carbides remain on the surface and their concentration drops with depth. It could be the first trace on the way to understanding the role of titanium in the ternary NEG. It is suggested by the Ti-Zr-V NEG’s lower susceptibility to ageing effects which, in the case of binaries, correlate with progressive cumulation of vanadium carbide (metallic vanadium acts as the reduction agent for zirconium during the activation) in the layer during air-venting – pumping cycles.

Bimetallic surfaces exhibit variety of catalytic properties which are interesting from the point of view of new catalyst designing. Reactivity of bimetallic alloys (transition metals in combination with metals having s,p- or even transition metal electronic configuration) can be significantly modified in comparison with pure metals.

In this study polycrystalline NiCu alloy surface was investigated by means of X-ray photoelectron spectroscopy (XPS), static secondary ion mass spectroscopy (SSIMS) and temperature programmed desorption (TPD). CO adsorption was measured both at room and low temperature on surfaces with different surface stoichiometry given by surface treatment (heating and ion sputtering). Results are compared with oxygen adsorption studies on the same sample.
ATOMIC PROBE STUDY ON STM TIP MODIFICATIONS

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In general, the chemical nature of the apex atom on the STM tip which provides the final states for tunnelling electrons and plays the key role in STM imaging is almost unknown. For a better understanding of the information provided by STM images comprehensive theoretical studies and model calculations have been performed [1]. The theoretical results support the view that in the most STM scans the tip is coated by atoms picked up at one stage or the other from the crystal surface [2]. To study experimentally modification on the tip due to the use in the STM, the UHV instrument was additionally equipped with an atom probe field ion microscope (AP-FIM) [3]. Before the use in the STM the tip can be characterized in the FIM. After use in the STM the tip can be inspected again by means of the field ion image. The chemical nature of species transferred to the tip apex has been determined by atom probe analysis. After reaching the jump to contact regime the transfer of sub-monolayer quantities of metal substrate atoms have been observed in the TOF spectra. Concerning the amount of matter transferred to the tip a strong influence of tip orientation and orientation of the substrate crystal was found in the experiments.

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STUDIES OF STRUCTURAL PROPERTIES ON MISMATCHED InGaAs/GaAs (001) HETEROSTRUCTURES BY XRD METHOD

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The heteroepitaxy of InGaAs/GaAs and related systems is of continuous interest not only for its technological importance but also because it serves as a model system of lattice-mismatched heterostructures that exhibit in-plane structural anisotropy. The asymmetry of the <110> in-plane directions manifests through several structural properties, such as different dislocation densities, anisotropic strain relaxation and morphological anisotropies. Several studies have been devoted to this topic using x-ray diffraction (XRD), transmission electron microscopy, electrochemical etching and other methods. In this work, we use high-resolution XRD to study the structural properties of highly mismatched InGaAs/GaAs (001) heterostructures grown by molecular beam epitaxy (MBE). The strain state and composition is measured along the two orthogonal <110> directions using the combination of symmetric and asymmetric rocking curves. Additional information is gained through reciprocal space maps. The results are compared to literature data. Our XRD studies indicate that thick, relaxed high misfit InGaAs/GaAs systems are nearly completely relaxed, and the unit cell
dimensions are isotropic. Anisotropy is present in the structural properties of the layers, caused probably by the substrate miscut, not by the A–B asymmetry.

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DEPENDENCE OF INTERFACE BROADENING ON LAYER THICKNESS.

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It is supposed that the best depth resolution in AES (or any other destructive) depth profiling of thin films with smooth free and internal surfaces can be achieved if ion sputtering of grazing angle incidence is applied. We have demonstrated, however, that if the sputter rates of neighboring layers are different then ion sputtering induced interface roughening might reduce the depth resolution. For this work we chose the Co/Cu system since the relative sputter rate is around 3.5 at angle of incidence of 85° [1]. Really in case of thick layers, 3XCo(30nm)/Cu(30nm), considerable interface broadening was found [2]. In this work we will report on AES depth profiling studies on layer system of Cu(10 nm)/Co(0.25 nm)/Cu (10nm)/Co(1.2 nm)/Cu(10nm)/Co(2.4 nm)/Cu/Si using 1 keV Ar⁺ bombardment with angles of incidence of 80° and 85° (with respect to the surface normal). At the given layer thicknesses interface broadening does not depend on the angle of incidence (at the studied angles). The results will be explained by showing that in the case of thin enough layers the mixed layer formed by ion bombardment is large enough to decrease the difference between the sputter rates of neighboring layers. This explanation will be supported by dynamic TRIM simulations as well.


DEVELOPMENT OF CONTROL SYSTEM FOR TWINMIC: NEW X-RAY MICROSCOPE FOR MULTIPURPOSE USE

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The potential of X-ray microscopy to probe heterogeneous phenomena at high spatial resolution is of great interest for a number of disciplines ranging from material science to biology, medicine, polymer science, geochemistry and environmental science. Two types of X-ray microscopes are currently in use – scanning and full-field imaging – with different but complementary imaging capabilities. A novel approach used in a project of the European Commission joining together eight partners is to integrate both microscope types in a single instrument with easy switch between the two modes. The instrument is optimized for the energy range of X-rays from 250 – 2000 eV and is currently situated at synchrotron light source Elettra in Italy. The microscope operates under high vacuum ambient while the specimen will be optionally situated also in air or inert gas atmosphere. The photon energy range covers the water window between the C, N and O absorption edges, L-edges of Fe, Ni, Co and K-edges of Na, Mg, Al, Si what will give the opportunity for advanced studies in biology, medicine, geochemistry, environmental and material research.
In this work we will present the basic set-up and the architecture of the control system of the new microscope. A user interface is developed that allows user-friendly control of the microscope station including (i) the precise movement of specimen, optical components and detectors in open and closed-loop operation, (ii) hardware synchronization and data acquisition, (iii) control of high resolution and fast read-out CCD-based X-ray detectors, (iv) control of vacuum level in different stages etc.

**ION BOMBARDMENT INDUCED DAMAGE ON SiC BY MEANS OF PLASMON LOSSES**

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SiC materials have a great importance in special semiconductor devices. Because the SiC is a compound material, the influence of an ion beam may result in composition change as well (beside structural damage). We have studied the effect of ion bombardment on the surface composition of SiC. It has been observed that Si/C ratio depended on the energy and type of ions, as well as the polarity of the crystal [1].

To better understand the ion beam influence on SiC, we have measured the elastic peak and the plasmon losses of ion bombarded samples in stationary condition. Some polytips of SiC (as 6H, 4H, cubic) have been irradiated by Ar ion beam with energy from 200 to 2000 eV. We found a broadening of the original plasmon peak, that can be decomposed for 2 components. One of the components can be assigned to the damaged layer, while the other to the undamaged one. The change of the plasmon energy can be interpreted by the change of the density and number of free electrons. The relative strength of the two plasmon intensities can be applied to determine the damaged layer thickness by the damaged material at the surface. It gives a tool for the estimation of the thickness of damaged layer.


**EFFICIENCY CALIBRATION OF ELECTRON SPECTROMETERS BY THE HELP OF STANDARD SPECTRUM**

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In the field of electron spectroscopic techniques the quantitative analysis is a very important application for studying thin films and surface nanostructures.

The most frequently used techniques are XPS, XAES [1,2] and AES in quantitative surface electron spectroscopy. In these techniques small changes in the detection efficiency vs. electron kinetic energy change the measured electron peak intensities and in this way the surface atomic concentrations. The importance of the precise determination of the atomic concentrations is very crucial, especially in the determination of non-destructive depth profiles by the help of AR-XPS
in which small changes in the concentrations can change dramatically the depth profiles of a few nanometer depth range.

In the present study the REELS technique was used to determine the relative detection efficiency by the help of standard spectrum measured on the surface of fine microcrystalline Cu specimen. The experimental studies were compared to theoretical calculations of the electron spectrometer efficiency.

The efficiency calibration is discussed from the point of view of quantitative XPS, AR-XPS AES and from the point of view of IMFP determination by XPS.

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References:

AES DEPTH PROFILING AND INTERFACE ANALYSIS OF C/Ta BILAYERS

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At present time much attention is paid on the synthesis and characterization of novel materials which are based on carbon in different chemical states, like graphite, carbide, carbonitride, hydrogenated carbon films, and composite materials on the basis of metal/polymer systems. Thin-film structures and composite materials are often depth profiled with one of the method for surface analysis in combination with sputter etching. However, many details of etching of carbon contained materials are still unexplained.

In this work, a bilayer structure of C-graphite(60 nm)/Ta(50 nm) was sputter deposited onto smooth-(111) substrates. To study the sputtering rates of carbon and Ta, the samples were AES depth profiled, applying 1keV and 3keV Ar+ ions, varying the angle of incidence in the range between 22° and 82°. The measured carbon concentration profile revealed a strong electron incidence angle dependent back scattering effect on the intensity of C(272 eV) peak. Depth profiles measured at different sputtering parameters were compared to theoretical depth profiles calculated by MRI-model (mixing-roughness- information depth) taking into account backscattering effect of primary electrons. It was found that the sputtering rates of C and Ta as well as ratio between them are strongly angle dependent. Depth resolution at the C/Ta interface was measured in dependence of different sputtering parameters on stationary and rotated samples. The results will be discussed in terms of structural changes induced on interfaces during AES depth profiling.
AN INVESTIGATION OF Fe$_{74}$Si$_{11}$B$_{14}$Ni$_{1}$ SOFT MAGNETIC POWDER

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The important characteristics of magnetically soft materials are their high permeability, high saturation induction, low hysteresis-energy loss and low eddy-current loss in alternating-flux applications. This group of materials includes high-purity iron, low-carbon iron, silicon steels, iron-nickel alloys, iron-cobalt alloys, and ferrites. Of particular promise are amorphous soft magnetic materials based on Fe, Si and B, which are usually produced in ribbon form using a melt-spinning technique. In our case, however, we prepared the Fe$_{74}$Si$_{11}$B$_{14}$Ni$_{1}$ powder using water atomisation, so that powder particles of less than 40 µm were obtained. The powders were then compressed together with a binding insulation material in order to obtain a SMC (Soft Magnetic Composite) material. The advantage of such a manufacturing process is the possibility of 3D motor design. The as-water-atomised powder particles were amorphous. Most amorphous alloys obtain their optimal electromagnetic characteristics when their microstructure is nanocrystalline, which means that the grain size is less than 100 nm. Therefore, the powders were exposed to different annealing temperatures and the microstructure changes were closely monitored. After 15 minutes of annealing at 500 °C dendritic structures appeared in the cross-sections of powders that were etched with 2% nital. Higher temperature annealing resulted in a two-phase nanostructured morphology. X-ray diffraction analysis showed these two phases were α-Fe and Fe$_{2}$B. The sizes of the grains belonging to these two phases were less than 100 nm, and this was confirmed with a TEM study. Additionally, the HRAES, Auger spectroscopy mapping and WDS were used for a nanoanalysis.

AES AND XPS INVESTIGATIONS OF MOLYBDENUM-SULPHUR-IODINE BASED NANOWIRE-TYPE MATERIAL

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Nanowire-type materials produced by single step synthesis were investigated by XPS, AES and HRAES. Sufficient quantity of these materials was produced for XPS analysis (approx. 2.5 x 1.5 mm$^2$ illuminated area). Large area AES analysis as well as corresponding analysis in selected spots or along selected lines on the surface of the bundles of the material were also performed. HRAES measurements on the bundles of the different thickness were performed and compared. All types of spectroscopy measurements were quantified especially with respect to Mo, S and I (which builds into the structure of the material during the synthesis). It was found that Mo:S:I stoichiometry rather deviates from the nominal one. It was also found that it is approximately constant along individual fiber but differs from one fiber to another. Thickness of the fiber seem to influence the sulphur content. Finally, stoichiometry obtained from XPS measurements differs significantly from the one obtained from the ARS measurements. Possible explanation
for this is somewhat larger probing depth of XPS compared to AES which would suggest radially inhomogeneous composition of the bundles.

MOLYBDENUM INTERLAYERS AS ADHESION PROMOTORS FOR THIN COPPER FILMS ON PLASMA TREATED GLASSY CARBON

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The adhesion of sputtered Copper (Cu) coatings on plane glassy Carbon (C) substrates can be increased by a Nitrogen-RF-Plasma pre-treatment and by use of thin Molybdenum (Mo) interlayers. As a model for the interface between Cu and C-fibers in Cu-C Metal Matrix Composites (MMC’s) the mechanical properties of the plane model system are of great interest. If the Cu-coating is deposited directly on a plasma-treated C-substrate at room temperature the adhesion of Cu to C is excellent. A following thermal treatment (800 °C, high vacuum, 1 hour) reduces the adhesion of Cu to C due to de-wetting of the Cu coating from the C substrate. De-wetting can be suppressed if a 100 nm Mo interlayer is present and the adhesion values are also increased again.

This work shall analyse the interface in the C/Cu and in the C/Mo/Cu samples by analytical Cross Sectional Transmission Electron Microscopy (X-TEM). At the C/Cu interface of Cu-films directly deposited on plasma treated substrates an intermixing zone between Cu and C of approximately 30-50 nm thickness could be detected. With Secondary Ion Mass Spectroscopy (SIMS) the presence of Nitrogen at the interface was clearly identified. After heat treatment the intermixing zone has vanished and also the Nitrogen content was significantly decreased.

Thermal treatment of the C/Mo/Cu sample shows no or only very little intermixing between Mo and C, while Cu and Mo obviously interdiffuse into each other. Additionally the formation of grain boundary grooves (responsible for the adhesion loss after thermal treatment) at the C/Cu interface is suppressed by the Mo interlayer.

This work is supported by the Austrian Science Fund (FWF) under grant Nr. P-14534. The TEM-investigations were performed in collaboration with the University Service Centre for Transmission Electron Microscopy (USTEM) of the Vienna University of Technology.
ON POROUS STRUCTURE OF Al-Cu ION-PLASMA FILMS


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Cathode sputtering and its modification magnetron sputtering promote the development of gas porosity in the condensates and so that the condensate density is proportional to the reaction gas partial pressure: sedimentation rate ratio.

Porous structures in ion-plasma friction-resistant Al-Cu-based coatings produced by the Bulat setup have vividly pronounced fractal properties. With their overall porosity of 16-18% the coatings possess the developed surface and owing to the system of communicating open pores provide efficient grease access to the zone of contact with friction surface.

It is evident that each separate pore is difficult if not impossible to individualize, therefore, submicropore (SMP) sizes can be measured only by convention.

Small-angle X-ray scattering (SAXS) – caused by highly disperse SMP in these coatings – is isotropic, asymptotic of SAXS indicatrixes \( \sim s^{-3.7} \) (s is a wave vector), and surface fractal dimension is \( D_{surf} = 2.3 \).

Along with it, the microanalysis and laser mass spectrometry data show that the coatings under study are characterized by high inhomogeneity of phase-elemental composition, which, in its turn, promotes pore formation.

DETERMINATION OF DOPING CONCENTRATION IN VERY THIN GaAs : Si LAYERS BY MICRO-RAMAN SPECTROSCOPY ON BEVELLED SAMPLES

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Doped nanostructures are attractive because of the interest in both an investigation of the fundamental physical properties and a fabrication of the electronic and optoelectronic devices. Determination of doping level in these structures causes difficulties in such well-known methods as C-V, classical Raman spectroscopy or Hall measurements.

In this paper we report a study of Si- doped GaAs layers prepared by MOVPE on Si GaAs substrates. Their thickness was 30 nm. Bevel were prepared from studied structures by chemical etching with very small angle (range of \( 10^{-3} \) rad). On these bevels micro-Raman study was performed by using of high power density of He-Ne laser light. The laser beam was focussed by microscopic lens system to a diameter about 1-2 \( \mu m \). The spectra were measured by back-scattering method at room temperature.
We observed high changes in ratio of TO and LO phonon intensities (TO/LO) by scanning of laser spot along the bevel. These changes were evaluated and discussed in the sense of coupling of photo-excited plasma of free carriers with LO phonon. The dependence of the ratio TO/LO was linear along the bevel length in depletion region. After calibration procedure its first derivative we estimated Si doping level of the doped GaAs layer in the vicinity of the interface UD GaAs/ GaAs : Si. Obtained values were compared with SIMS measurements.

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BULK AND SURFACE CHARACTERISTICS OF METAL POWDERS FOR DIRECT LASER SINTERING

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Nowadays, Direct Metal Laser Sintering (DMLS) of metal powders is already a well-known and established process for fast and effective manufacture of prototypes and tools for injection moulding of polymer-based products (PIM) in smaller series. However, the final goal is to improve the quality of DMLS products (tools) in order to enable production of larger series of PIM products or even to produce tools which can sustain more rigorous conditions during pressure die casting of Al-based or other non-ferrous alloys. Therefore, permanent improvement of equipment, as well as metal powders for DMLS is necessary.

Generally, users of DMLS equipment and producers of tools or prototypes are not well acquainted with metal powder characteristics and mechanisms of their sintering. Therefore, they primarily depend on information of DMLS equipment producers, which usually deliver also DMLS powders. In Slovenia tooling for PIM and pressure die casting of non-ferrous alloys has a long tradition and is well developed. The new equipment for DMLS, purchased and installed at RTCZ Hrastnik, has enabled Slovenia to make also a breakthrough in the field of rapid prototyping and manufacture of DMLS prototypes and tools. This demands good knowledge of metal powders’ characteristics, of the possible mechanisms of their sintering, as well as of the characteristics of the sintered products.

In the frame of the present contribution, two different (Cu and Fe-based) metal powder mixtures for DMLS were analysed. Bulk chemical compositions were determined with ICP-AES. Morphological, as well as microstructural properties of individual metal particles were determined with the light and SEM/EDS microscope. Additionally SEM/EDS and WDS surface mapping has been performed with the new high-resolution field-emission Jeol JSM 6500F for surface analyses of powder particles on the nano level.
XPS ANALYSIS OF PULSED PLASMA ION NITRIDED Ni- AND Ti-ALUMINIDES

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The complex nitrides of intermetallics based on titanium, aluminium, nickel and iron are very prospect materials for the high performance coatings. Samples of Ni₃Al and TiAl₃ were prepared by the Self-Propagating High-Temperature Synthesis (SHS) of elemental powders in argon atmosphere, followed by pulsed plasma ion nitriding. Samples were evaluated by metallography, hardness measurements. Results on combustion synthesis of aluminides of nickel and titanium, followed by ion nitriding are presented. X-ray photoelectron spectroscopy (XPS) analysis of Ni₃Al confirmed formation of nitride with binding energy Al2p 74.6 eV, formation of AlN on both aluminides, but no presence of TiN nitrides was confirmed on TiAl₃.

SEM INVESTIGATION OF BACTERIA DEPOSITED ON ALUMINUM FOIL

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Two types of bacteria used as sterilization indicator has been prepared in liquid solution: Bacillus subtilis and Bacillus stearothermophilus. Bacteria have been left in an incubator at appropriate temperature for 4 days in order to allow for spore formation. The liquid solution rich in spores have been applied to carefully cleaned aluminum foils. The solution was dried in air under prescribed conditions for 24 hours. The samples were covered with a thin gold film by sputter deposition and mounted into a Scanning Electron Microscope. The bacteria were imaged using a low-voltage and current beam of electrons in order to prevent radiation degradation of the spores. It was found that the Bacillus stearothermophilus has dried in the form of perpendicular lines, while the Bacillus subtilis has dried in well – defined clusters. Radiation damage of the bacteria was investigated in situ at a high voltage and current electron beam. It was found that the application of the beam caused slow degradation of bacteria.
TIN OXIDE THIN FILMS STUDIED BY SECONDARY ION MASS SPECTROMETRY (SIMS) AND ATOMIC FORCE MICROSCOPY (AFM)

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The SnO2 thin films are widely used for gas sensor application. The gas sensing properties of the films are strongly dependent on the morphological properties, like surface roughness, grain size, grain geometry, and chemical composition. The material properties depend on the preparation procedure. In this contribution we have studied the properties of tin oxide films prepared by plasma oxidation of thin tin films.

Our previous measurements indicate that the oxide films prepared by plasma oxidation contain a mixture of SnO, and SnO2 oxides. Therefore we prepared a set of samples oxidized with different oxidation times at higher substrate temperature (200 °C).

The SIMS depth profiles were measured by ATOMIKA 3000 SIMS system. The SIMS analysis was made using a Cs+ ion gun. The primary ions of an energy 3.3 keV and a current 80 nA were rastered over an area of 0.5 mm × 0.5 mm. The surface topography and roughness were measured by the AFM (Metris – 2001A – NC, Burleigh Instruments Inc.) All AFM measurements were carried out in the contact mode at the ambient atmosphere and at room temperature. Scan was made over areas from 1.2 µm × 1.2 µm to 25 µm × 25 µm.

The surface analysis of tin oxide thin films indicate that the parameters of preparation procedure play a role in determining the surface topography and stoichiometry of tin oxide films prepared by plasma oxidation. It has been shown that the method of plasma oxidation is applicable for preparation of SnO2 thin films for gas sensors.

SOME ASPECTS TO THE RHEED DECAY BEHAVIOUR OF LT-GaAs GROWTH

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Recently, molecular-beam-epitaxy (MBE) growth of GaAs at low temperature (LT) – around 200 C – has an increasingly importance in the semiconductor research and technology. The LT-GaAs growth has become an expanding important method since it provides highly insulating films and contributes to the synthesis of magnetic semiconductors. It was shown that growth at this LT leads to incorporation of excess As in the crystal. The high concentration of excess As in LT-GaAs results in a number of novel properties. The RHEED behaviour during MBE growth on GaAs (001) surface under LT growth conditions is examined in this work. The reflection high-energy electron diffraction (RHEED) and its intensity oscillations of LT-GaAs growth have some particular behaviour. The intensity, phase and decay of oscillations depend on the beam equivalent pressure (BEP) ratio and substrate temperature etc. We examine here the intensity dependence of RHEED behaviour on BEP ratio, substrate temperature and the excess of As content in the layer. The change of the decay constant of the RHEED oscillations is also
discussed. The decay and absence of the RHEED intensity oscillations can origin from several 
effects e. g. change of sticking coefficients, change of unperturbated area and change of strain. 
Here was found that the separation of growth and strain influence on the RHEED oscillation 
decay in the case of LT-GaAs is possible in a narrow region of BEP ratio.

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AES DEPTH PROFILING OF IRON-OXIDE LAYERS

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In the field of electrotechnical engineering there is an interest for substitution of enamelling 
procedure with controlled oxidation of electrical steel sheets for the formation of isolation and 
protection layer. The property of steel sheets depends on the thickness of oxide layer, the 
chemical state of elements and the ratio between different iron oxides. Routine AES depth 
profiling allows insight into the depth distribution of elements, while the chemical state of 
different chemical species inside the layer is often unknown.

In this work we will present an application of AES depth profiling for investigation of different 
oxide layers prepared with technological process on non-oriented electrical steel sheets by 
controlled oxidation in gas mixture, which followed recrystallization and decarbonization 
annealing processes. Fe-oxide layers with different quality level of adhesion to the steel sheet 
were obtained by varying parameters like temperature and time of heat, composition of gas 
mixture, surface cleaning, etc. Comparison between AES depth profiles obtained on the 
oxidized layers of steel sheets and those obtained on reference oxide samples, allowed us to 
determine the depth distribution of different types of iron oxides in the oxidized layers. The 
composition of oxide layers was correlated with parameters of technological process and quality 
of adhesion between oxide layers and substrates.

DIMENSION OF ETCH PIT ON GaAs (001) SURFACE AFTER 
ELECTROCHEMICAL TREATMENT

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The selective chemical and electrochemical methods have been long known and widely used as 
examining and processing techniques, especially in the compound semiconductor technology 
for a long time. The selective etching is suitable for the development of dislocations and other 
crystal imperfections. Several authors have dealt with revealing of the defect on GaAs (001) 
surface using nonpreferential etching.
The dimension of the etch pits will determine quantitatively in our work. An electrochemically etched surface have served as an experimental basis for the investigation. The explanation of crystal decomposition is operated by biatomic layers. The decomposition by molecular manner is verified using an independent measurement. The formation of the etch pits shape was simulated on a lattice node model where the sequence of the bond breaking is taken into consideration. The reciprocal value of the relative bonding energies is connected with the probability of the breaking of two and three dangling bonds As-Ga molecule. The dimension of the simulated object correspond very well with the pits developed on electrochemically etched GaAs (001) surface.

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**SOME ASPECTS TO THE INTENSITY MEASUREMENT OF RHEED**

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The growth of perfect crystal layers and low dimensional structures needs the control of epitaxy. The RHEED pattern and its intensity oscillation carry in-situ information for us about the crystal growth. The getting of growth information from the reflected – diffracted pattern, we need the understanding of the interaction between the grasing incident electron beam and the growing crystal surface. RHEED is a widely used monitoring technique during MBE growth. The orientation, quality and reconstruction of the grown surface can be determined by the RHEED pattern. The intensity of the RHEED pattern oscillates under appropriate conditions during the growth process. The growth rate, and the composition in the case of alloy materials can be determined with the help of RHEED oscillations. The InGaAs/GaAs and also InAs/GaAs heterostructures are of a great importance not only in the semiconductor technology but because they serve as modelsystem for the investigation of lattice mismatch growth and of QD formation. The study of the damping of the RHEED intensity oscillation is a possible and useful in-situ technique to observe 2D-3D transition of the grown layer. We will be shown the information which is carried by the intensity damping. The RHEED pattern and intensity of oscillation are widely used by video camera focused on the fluorescent screen of MBE chamber. The fluorescent film on the window and the camera have surely non-linear behaviour in the intensity, which can deform the observed intensity distribution. The direct measurement of electron current is more advantageous than the indirect mode but its application and adjustment are more cumbersome. If we want to investigate real intensity decay of the oscillation we must measure the electron current directly.

In our work, the growth of InGaAs layers on GaAs (001) substrate was investigated, which is carried out by MBE at 490 °C deposition temperature. In this case the accurate intensity of RHEED oscillations was measured directly by means of high resolution Faraday cup. This cup had three pinholes along a line of decreasing diameters in order to obtain good angular resolution and suppression of the electron background. The cup was attached to a precision manipulator. The current was measured with an electrometer. We have received exact exponential function
for the decay with the help of direct measurement of electron current. This results give possibility to estimate the crilical layer thickness from the decay of oscillation.

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DECOMPOSITION OF CH₄ ON TiO₂(110) SURFACES OF DIFFERENT STOICHIOMETRY AND NOBLE METAL DECORATION

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The conversion of saturated hydrocarbons into more valuable compounds is one of the most important reactions in the field of heterogeneous catalysis. However, the identification of the surface sites responsible for the catalytic conversion of CH₄ (C-H breaking, C-C coupling) is far from a complete understanding. The present work is devoted to this question on a special catalytic 2D model support TiO₂(110). The main methods applied in this work: scanning tunnelling microscopy-spectroscopy (STM-STS), Auger-electron spectroscopy (AES) and thermal programmed reaction (TPR).

The TiO₂(110) substrates used in the present study exhibited several different surface morphologies: (i) large, well ordered (1x1) terraces decorated by some additional [001] rows of a reduced TiOₓ phase; (ii) strongly defected surface produced by low energy Ar⁺ treatments; (iii) ordered surfaces decorated by noble metal nanoparticles of different sizes and distributions. All of these surfaces were tested in the decomposition of CH₄ at 10⁻¹ mbar.

The experiments have shown that the nanostructure of the carbon layer formed in the decomposition of CH₄ at 600 K temperature is different for the admetal-free and Rh (or Pt) covered support: characteristic carbon-clustering was observed for the latter cases. The freshly Ar⁺ treated TiO₂(110) surface exhibited very high activity, especially in the presence of noble metal adparticles. The measurements performed by a mass spectrometer revealed that the gases formed during the decomposition of CH₄ were mainly CO₂ and H₂O, suggesting a complete oxidation of the surface intermediates besides the accumulation of carbon nanoclusters.

FORMATION OF DIAMOND NANOCRYSTALS BY CARBON IMPLANTATION

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We investigate the formation of diamond nanocrystals, produced by ion beam implantation of carbon in fused SiO₂ substrates, followed by thermal annealing. Two annealing techniques, Rapid Thermal Annealing (RTA) for 100 seconds, and furnace annealing for one hour, are performed under forming gas atmosphere for three different carbon doses. This allows us to study the formation of diamond nanocrystals and other carbon phases. Optical Absorption and Raman Spectroscopy measurements show formation of carbon nanocrystals in diamond (sp³ bonded) and graphite (sp² bonded) form depending on the dose and the annealing method.
INFLUENCE OF CATALYST PRETREATMENT ON CARBON NANOTUBES GROWTH

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There are three basics methods to produce carbon nanotubes: the well-known arc discharge and laser ablation and promising chemical vapor deposition (CVD). In recent years, research has focused on developing CVD methods, because they promise to produce high quality nanotube materials at large scales. Moreover, controlled CVD growth strategies on catalytically patterned substrates yield ordered nanotube architectures and integrated devices that are useful for fundamental characterizations and potential applications of nanotube molecular wires. Catalytic patterning combined with CVD growth represents a novel approach to ordered nanowire structures that can be addressed and utilized.

Growth of carbon nanotubes and their properties are significantly affected by type, thickness and texture of catalyst and catalyst-support interactions. The relationship between carbon nanotubes density and thickness of catalyst as well as different growth behavior of CNT on various supports is known.

This work studied influence of annealing in vacuum and NH₃ pretreatment on subsequent growth of carbon nanotubes by alcohol catalytic CVD method. The samples used in our experiments were Si (100) wafers covered with thin Ni layer by magnetron sputtering. The effect of annealing in vacuum and different time NH₃ pretreatment on CNT density is observed. Raman spectroscopy and Scanning Electron Microscopy have been used to provide further data on the nature of the nanotubes grown on these samples.

ANALYSIS OF THE NANO-STRUCTURAL PROPERTIES OF AMORPHOUS HYDROGENATED SILICON-CARBON ALLOYS

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Amorphous hydrogenated silicon-carbon thin films were deposited by sputtering of silicon target in gas mixture containing hydrogen and carbon atoms, with extensive variation in hydrogen content and carbon to silicon ratio. The composition of specimens and theirs structural ordering on atom level was estimated by IBA (Ion Beam Analysis), e.g. RBS (Rutherford Back Scattering) and ERDA (Elastic Recoil Detection Analysis), FTIR (Fourier Transform Infrared) and Raman spectroscopy. The spectral distribution of dielectric function was estimated upon standard transmittance and reflectance measurements in uv-visible part of the spectrum.

The homogeneity of deposited films on nano-scale was examined by GISAXS (Grazing Incidence Small Angle X-ray Scattering), performed on ELETTRA synchrotron radiation source, Trieste (Italy). Results of GISAXS spectra of all of measured specimens indicate
presence of "particles" in the "bulk" of the films, with size distribution between 1 and 2 nm and mean values variation between 1 and 6 nm.

In order to determine the constitution of the "particles", the specimens were modelled as a multy-component material with certain dimensions and certain optical properties for each of component, considering the results of all of applied methods. The possibilities of this kind of analysis have been discussed on several typical examples.

DIAMOND AND DIAMOND-LIKE CARBON CVD FILMS ON Si, TiN, WC-Co AND Al₂O₃ SUBSTRATES

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On several examples, we have demonstrated the potential of Raman spectroscopy and SEM to identify the state of the film-to-substrate interface after single technological steps. We have resolved diamond and non-diamond phases of carbon. A serious problem in the use of polycrystalline CVD coatings in electronics, optics as well as in cutting tools is the surface roughness. Nanocrystalline and microcrystalline types of diamond have their typical Raman spectra. The Raman spectra contain, besides peaks of various phases of carbon, also a luminescence band. Raman measurements were conducted on a Raman spectrometer DILOR-JOBIN YVON-SPEX, type LabRam. Scanning electron microscopes Leo 1550 and Philips 505 operating in the secondary electron mode were used to study the microtopography of diamond layers.
Rigorous coupled wave analysis has been used to predict appropriate parameters for fabrication of wire grid polarizers (WGP) requested for visible and near-ultraviolet (UV) spectral region. The formulae derived within this theoretical approach were employed for characterizing three samples of the WGP created on the basis of TaN. These formulae were namely used to treat the experimental data obtained by measuring the spectral dependences of the ellipsometric parameters in reflected light corresponding to specular direction (i.e. the zeroth reflected diffraction order). Using this treatment of the experimental data the values of the grating parameters, i.e. the values of the depth a period, were determined for all the three samples. The spectral dependences of the optical constants of TaN were evaluated using the TaN films deposited onto SiO₂ substrates under the same technological conditions as the gratings. Further will be shown that the gratings studied are relatively good polarizers in the visible region while within the near-UV they are not being the sufficiently good ones. This fact will be proved by both the theoretical and experimental ways within spectroscopic ellipsometry applied in the transmission mode. Furthermore it will be shown that the WGP also represent the sufficiently good polarizers for the near-UV region if metals are used to fabricate them.

Due to their novel physical properties, one-dimensional (1-D) nanomaterials are supposed to have potential applications to nanoelectronics and optoelectronics. Since gallium oxide (Ga₂O₃) is a stable wide-bandgap compound with intense luminescence properties, researchers have studied on the growth of Ga₂O₃ nanostructures by various techniques. In this paper, we have successfully synthesized the gallium oxide (Ga₂O₃) nanomaterials such as nanorods, nanobelts, and nanosheets by the thermal evaporation of GaN powders. We have investigated the effect of metal catalyst in growing the nanomaterials. X-ray diffraction, scanning electron microscopy, transmission electron microscopy, and energy dispersive x-ray spectroscopy were used to characterize the structure, morphology, and composition of the samples.
ORIENTATION AND PACKING OF KEGGIN-TYPE ALUMINATE ARRAYS; AN STM STUDY

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The so-called Keggin-ion, \([\text{AlO}_4\text{Al}_{12}\text{(OH)}_{24}(\text{H}_2\text{O})_{12}]^{7+}\) ion is a result of the forced partial hydrolysis of AlCl₃ solution. In the form of sulfate or selenate salts, its structure was resolved long ago [1]. This and related polyoxo-kations as well as polyoxo-metalate-anions have similar structures named "Keggins". These discrete nano-scale clusters are promising candidates for shape selective catalytic or sensor applications [2].

Samples for STM imaging were prepared by depositing one drop of aqueous Keggin-ion solution onto freshly cleaved highly oriented pyrolitic graphite (HOPG) surface [3]. After drying, a polycrystalline rind at the perimeter of the original droplet encloses a large region where the deposit is thinner and STM images can be obtained. This region may exhibit small patches, oriented rod like crystalline as well as bar HOPG. The oriented crystalline roods consist of more, often twin structured, parallel line which are approximately 1 nm in high above the flat region. This finding can be interpreted as a germ of Keggin-ion sulfate, which is consistent with its macroscopic structure.

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GIANT MAGNETORESISTANCE IN EVAPORATED Fe/W MULTILAYERS

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Giant magnetoresistance (GMR) was broadly studied in multilayers (MLs) composed of magnetic layers and Cu or Ag spacers. An advantage of these combinations is their immiscibility, i.e. stability of interfaces. Low miscibility is expected also in Fe/W couple, where the heat of formation of compounds is \(\Delta H = 0\). Therefore, GMR in this type of ML was studied here. MLs were UHV e-beam deposited onto Si, number of periods being 10. Temperature of substrate was between RT and 200 °C. Layer thickness of Fe and W was 1 nm or 2 nm, resulting in four different designs. Samples were analyzed by X-ray diffraction, X-ray reflectivity and X-ray diffuse scattering. GMR was measured in the field \(\leq 0.5\) T. In RT deposited samples GMR ratio was \(\leq 0.15\%\) even at 4.2 K. GMR enhancement was achieved by deposition at 190 °C. Here, the GMR ratio was 2 % already at RT. According to X-ray examinations RT deposited samples are amorphous. At 190 °C structural coherency and strong lateral and vertical interface roughness correlation was found. E.g. lateral and vertical correlation lengths are 9 nm and 25 nm.
Carbon nanotubes were grown on the filaments of light bulbs in 10 ml.s⁻¹ Ar containing 0.1 and 0.2 ml.s⁻¹ C₂H₂ at 700-900 K. The SEM micrographs show the variation of the characteristics of the tubes with the test temperatures and the C₂H₂ potentials. The EDX point analysis on the tubes shows the existence of carbon. Electron-field emission of nanotubes grown on the filaments was measured using the applied voltage of 250 V. It was found that the current was controlled by the applied voltages and the test temperatures.

Keywords: Carbon nanotubes, Electron-field emission.

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RHEED STUDY OF SUPPORTED Pd-Sn BIMETALLIC NANOCLOUDERS

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In the recent years, a great effort has been done to investigate the structural, electronic and chemical properties of bimetallic systems. It is well known that a bimetallic surface can exhibit chemical and catalytic properties that are very different from those of the individual metals. Large effects are observed for bimetallic surfaces that combine a metal with an electron-rich d band (late transition metal) and a metal with an electron-pure valence band (early-transition or sp metal). The XPS (X-ray Photoelectron Spectroscopy) investigation of Pd/Sn alloy indicated a strong bimetallic interaction resulting in a noble metal-like electronic structure.

Pd/Sn bimetallic thin films were grown by molecular beam epitaxy on different single-crystalline non-conductive substrates. Substrate and deposit crystallographic structures and evolution of deposit lattice parameter during the growth were studied by reflection high-energy electron diffraction (RHEED). Intensity timescans and evolution of intensity profiles of diffraction spots gave information on the formation of bimetallic system. The early stages of the growth in dependence of Pd and/or Sn deposition sequence were studied. The results show that the growth mechanism depends on the deposited metals succession. The formation of epitaxial nanocrystals was observed. The reflection high-energy electron loss spectroscopy (RHEELS) was used as an auxiliary method for chemical analysis.
FORMATION OF SI NANOPARTICLES AND SIO₂ CRYSTALLITES BY THERMAL ANNEALING OF SIOₓ FILMS

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Amorphous silicon oxides (SiOₓ) films have found a new area of application in connection with the possibility of obtaining Si nanoclusters embedded in SiOₓ matrix as a result of heat treatment. Appearance of photoluminescence (PL) bands related to the formation of Si nanocrystallites in annealed SiOₓ films has attracted wide interest due to potential application of such material in Si-based optoelectronic devices.

In this paper results from IR spectroscopy, spectral ellipsometry (SE) and X-ray diffractometry (XRD) investigations of thin SiOₓ films are presented. The SiOₓ layers were deposited by thermal evaporation of SiO in vacuum. The Si substrate temperature during deposition was 150 °C. In order to obtain amorphous and crystalline Si inclusions in the oxide matrix thermal annealing was performed in Ar atmosphere for 5 and 30 min at 700 and 1000 °C.

Analysis of the IR and SE spectra of the films has shown that annealing leads to compositional change of the film, formation of Si nanoclusters and densification of the films, expressed in the increased value of the refractive index. XRD spectra detect crystalline SiOₓ inclusions with a hexagonal structure. Conclusions can be made that the amount and size of SiO₂ crystallites depend mainly on the duration of annealing rather than on temperature.
MECHANICAL PROPERTIES OF Al-Cu-Fe THIN FILMS

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A series of Al/Cu/Fe multilayer films was deposited by sputtering of elemental targets. The depositions were distinguished by the following parameters: individual layer thickness, total thickness (i.e., number of stacks), layer sequence and overall composition. Most of the tests were conducted on tool steel and hard metal substrates. In order to ensure proper homogenization of the film, the samples were heat-treated in a tube furnace with argon flow. Different annealing patterns were tried with an emphasis on the dependence of mechanical properties on annealing temperature. The composition was chosen as such to get the β-Al(Cu,Fe) phase, which is an approximant of the icosahedral phase Al\textsubscript{62.5}Cu\textsubscript{25}Fe\textsubscript{12.5}.

The scratch-test technique was used to evaluate the adhesion of the film as well as the scratching coefficient. There were substantial differences in the scratching force behavior using different annealing temperatures. The lowest scratching coefficient and the highest critical load were obtained in the annealing range of 500–600 °C. Microhardness was measured by Vickers microindentation in a load range of 5–100 g. An increase of a factor of 2.5 was obtained for heat-treated films, with a plateau being reached above 400 °C. Among other properties, surface roughness and thickness were studied.

XRD ANALYSIS OF TUNGSTEN THIN FILMS

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The properties of dc-magnetron sputtered thin tungsten films depend very strongly on deposition conditions. Depending on Ar gas pressure, substrate material, substrate temperature and deposition duration, there is a different relative amount of beta- and alpha-W phase deposit. In this work tungsten thin films prepared under different conditions of working-gas pressure and deposition temperature were investigated by means of X-ray diffraction (XRD) on a Philips powder diffractometer (PW 1820) and subsequently refined by the Rietveld method. The goal of this work was to investigate the dependence of microstructure and phase composition on deposition condition. Moreover, the microstructure, size and microstrain were extracted by different methods: Rietveld method, single-line method and Warren-Averbach method. The structures were refined in the space group of α-W, Im-3m and β-W, Pm-3n. The quantitative phase composition was estimated from refined values of scale factors according to the procedure of Hill and Howard. It was found that at the deposition temperature of 293 K, on increasing the working-gas pressure, the amount of β-W phase increases. However, at low (77 K) and high (523 K) deposition temperature, on increasing the working-gas pressure, there is a sudden decrease in the amount of β-W phase at 1.4 Pa, then a further increase of working-gas pressure yields to the increase in the amount of β-W phase. The lattice parameters of alpha and beta...
tungsten film decrease with the increasing of working-gas pressure. The results of size-strain analysis, volume averaged grain size and r.m.s. microstrain obtained by different methods used in this work are in good agreement.

SOLID STATE SURFACE WETTING OF TIN ON ALUMINIUM UNDER UHV CONDITIONS

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If Tin (Sn)-islands exist on a bare Aluminium (Al)-surface, Sn forms a monoatomic wetting layer on Al under Ultra High Vacuum (UHV) conditions. The islands act as Sn-reservoirs and emit Sn until a monolayer of Sn is formed on the surface. Experimentally this process was investigated by Scanning Auger Microscopy (SAM). The over-all thermodynamic driving force is the reduction of the surface energy of the bare Al-layer by the total coverage with Sn.

In this paper the mechanisms leading to wetting layer formation will be discussed in more detail by means of thermodynamic considerations and Kinetic Monte Carlo Simulations (KMC). Thermodynamics is employed to describe the lateral coverage of the free Al surface by mass emission from pre-existing Sn-islands. The Surface energy of the bare surface is considered higher than the surface energy of the totally covered system. KMC, on the other hand, will be employed to describe the dynamics of the process and to understand the fact why the thickness of the wetting layer is limited to one monolayer. From a comparison with experimental data from SAM measurements the microscopic diffusion parameters which are involved in the formation of the wetting layer are qualitatively identified.

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CHARACTERISATION AND SURFACE ANALYSIS OF CVD IRON

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Growth of pure iron thin-film by chemical vapour deposition (CVD) from iron pentacarbonyl (Fe(CO)_5) liquid source precursor is reported. Thin iron layers were deposited in a low-pressure process chamber using a radiantly heated graphite susceptor on 200nm thick silicon dioxide layer with hydrogen as carrier gas for the precursor into the chamber and as removal of reaction by-products. Deposition without carrier gas was found to produce inconsistent layers. Iron thin films with body-centred-cubic (bcc) structure were deposited at various temperatures between 200 to 300 °C with precursor flows above 6 sccm, and hydrogen flow at 500 sccm at 1.5 torr. Film thicknesses from 115 to 175 nm were achieved for different process conditions. Material
characterisation were carried out to study the effect of key process parameters on film resistivity, purity (by x-ray diffraction analysis), surface roughness (by atomic force microscopy) and effects of high temperature rapid thermal annealing (RTA). Initial x-ray diffraction analysis shows \(\alpha\)-Fe 110 bcc structure. Resistivity of deposited film, 120 \(\mu\Omega\cdot\text{cm}\), was found to be 10 times higher than that of bulk values. Material resistivity was found to be decreasing with annealing temperature. For a 172 nm thick layer, film resistivity drops to 55\% (65 \(\mu\Omega\cdot\text{cm}\)) of its initial value after annealing at 700 °C. Surface roughness of the iron layer before and after annealing is 16 nm and 22 nm respectively. Surface and structural analysis of the film were conducted with a thermal field emission scanning electron microscope (SEM). Measured grain size is less than 150 nm and the grain structure changes significantly when subjected to high temperature annealing.

**INVESTIGATION OF SPUTTERED AMORPHOUS SILICON AND SILICON OXYNITRIDE MULTILAYERS**

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Sputtered SiO\(_x\)N\(_y\) (SiON) films have several applications in microelectronics and optoelectronics, such as passivating coatings, thin gate dielectrics and optical waveguide or antireflection coatings.

By depositing alternating dielectric SiON and amorphous silicon (a-Si) layers with thickness in the nm range on single crystalline silicon, a multilayer structure with a new type of photoluminescence behaviour can be obtained. Emission of visible or near infrared light yields physical properties of quantum-well-like structure. The dependence of the optical emission on the thickness of a-Si layer should be related to the conduction and valence band shifts due to the quantum confinement effect. RF sputtering using Ar\(_2\) or N\(_2\) plasma can deposit layers with thickness of some nanometers. In this paper we deal with the quality assessment of SiON/a-Si layers deposited by sputtering.

The layers were sputtered from semi-insulating Si or phosphorus doped Si target in a conventional high vacuum apparatus with base pressure better than 5·10\(^{-5}\) Pa. Single wavelength and spectroscopic ellipsometry and cross-sectional transmission electron microscopy (XTEM) were applied for studying the multilayer structures. Good agreement was obtained between results extracted from XTEM and ellipsometry concerning thickness determination.

Better interface quality was observed between the layers sputtered from phosphorus doped Si target. This can be due to the incorporation of the sputtered P atoms into the growing SiON layers. Since diffusion of P in silicon oxynitride is suppressed, the sputtered P atoms do not diffuse to the substrate but remain in the SiON layers. As P occupies substitutionally Si sites, its incorporation creates Si interstitials in the SiON layers, which is not the case when a semi-insulating Si target is employed. Such excess Si interstitials and their diffusion on the SiON layer surface may allow for the optimised rearrangement of the incoming O and N atoms with the elimination of kinks and steps and achievement of smoother surfaces with respect to the case of the semi-insulating Si target.
OXIDATION BEHAVIOUR AND THERMAL STABILITY OF Al<sub>1-x</sub>Cr<sub>x</sub>N HARD COATINGS

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Recently, wear-resistant coatings within the system Al-Cr-N consisting of the metastable face-centred cubic Al<sub>1-x</sub>Cr<sub>x</sub>N phase have been successfully introduced to the market. The aim of this work was to evaluate the oxidation resistance and the thermal stability of these coatings in dependence of the Al content. Al<sub>1-x</sub>Cr<sub>x</sub>N coatings with different Al content have been grown by reactive cathodic arc evaporation. The oxidation resistance was investigated using thermo-gravimetric analysis (TGA) and annealing experiments in oxidizing atmosphere. Differential scanning calorimetry (DSC) up to 1450 °C in argon and oxygen-containing atmospheres was used to study the resistance against decomposition of the metastable Al<sub>1-x</sub>Cr<sub>x</sub>N phase. The activation energies of the occurring reactions such as recovery, decomposition or oxidation were determined using different heating rates in the DSC experiments. XRD and TEM investigations were carried out to investigate the phase changes due to the different heat treatments. It was found that during TGA measurements in inert atmosphere the CrN phase starts to decompose at 850 °C and transforms to pure Cr by passing the Cr<sub>2</sub>N phase. These two reaction steps are parallel and connected with a release of N<sub>2</sub>. High aluminium contents increase the onset of the decomposition effect by more than 200 °C. During annealing in oxidative atmosphere in the same temperature range, no evidence for decomposition of the CrN phase was found.

INFLUENCE OF ADDITIONAL ION BOMBARDMENT BY NITROGEN ON CHARACTERISTICS OF DUPLEX COATINGS

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A duplex surface treatment involves the sequential application of two surface technologies to produce a surface composition with combined properties. A typical duplex process involves plasma nitriding and the PVD coating treatment of steels. In the paper are presented characteristics of hard coatings, type TiN, produced by classic technology PVD (physical vapour deposition) and IBAD (ion beam assisted deposition). Subsequent ion implantation was provided with N<sup>+</sup> ions.

The film deposition process exerts a number of effects such as crystallographic orientation, morphology, topography, densification of the films. The optimization procedure for coated parts
could be more effective, knowing more about the fundamental physical and mechanical properties of a coating and their interdependence. The effects on the structure as well as mechanical properties of the films were investigated in detail in the present research. A variety of analytic techniques were used for characterization, such as scratch test, calo test, SEM, XRD and EDAX. The results were correlated with properties determined from mechanical and tribological characterization. Therefore, by properly selecting the processing parameters, well-adherent TiN films can be obtained on engineering steel substrates, and show a potential for engineering applications.
**EFFECT OF NITRIDATION AND CARBURIZATION OF γ-TiAl ALLOYS ON WEAR RESISTANCE**

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The 1,000 – 1,300 K nitridation and carburization processes of MJ12 (Ti-47Al-2Nb-2Cr) and MJ47 (Ti-47Al-2Nb-2Mn+0.8TiB₂) were studied in the purified ammonia and acetylene. Flow rate of the ammonia was kept constant at 10 cm³.s⁻¹ but that of the acetylene was kept at 0.01, 0.03 and 0.05 cm³.s⁻¹. The properties of the alloys were extensively analyzed. The Knoop hardness values were increased with an increase in the temperatures and acetylene potentials. A WC pin-on-disk wear rate was reduced by one or two orders of magnitude in comparison with the untreated alloys. In addition, the surfaces of the alloys were characterized using the XRD, SEM and EDX analyzers to determine the new phases formed.

Keywords: γ-TiAl, Nitridation, Carburization, Tribological test

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**KINETICS OF STRUCTURAL RELAXATION OF AMORPHOUS AlW THIN FILMS UNDER ISOCHRONAL CONDITION**

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Structural relaxation of Al₈₀W₂₀, Al₇₈W₂₂ and Al₇₅W₂₅ amorphous thin films under isochronal condition was examined by continuous in situ electrical resistance measurements in vacuum. The amorphous AlW thin films were prepared by simultaneous dc sputtering of both pure aluminum and pure tungsten by two independently controlled magnetron sources. For the isochronal heating, it was observed that the relaxation effects decreased with an increase of the heating rate and decreased with the tungsten content in the film. Inspection of the resistivity versus temperature data suggests that the relaxation kinetics can be described by the phenomenological JMA kinetic law for isothermal transformation. The adaptation of the JMA model to the non-isothermal kinetics was used for calculation of kinetic parameter n. The calculated results indicated that the kinetic parameter n in AlW amorphous thin films increases from approximately 0.4 to 0.8 with increasing of the aluminum content. The results suggests that the relaxation of the AIW amorphous structure changes of interatomic distances and thus increases the chemical short range order (CSR). Due to sp-d hybridization the change in interatomic distances strongly affects the electrical properties of AlW amorphous alloys.
INTERFACIAL REACTIONS IN Si/Co METALLIZATION CONTACTS DURING HEAT TREATMENT

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The increasing interest in Co-Si intermetallics is driven both scientific and technological concerns. Cobalt silicides attract much attention because of their possible application in VLSI technology as an interconnect material. Understanding and control the silicide formation to obtain desirable metallization is therefore paramount. Experiments were designed to determine phase equilibria in the Si-Co binary system in case of thin film samples. Microstructural evolution of the reaction zone has been studied in thin bi- and multilayered diffusion couples. Three different cases were studied, in which the thin Co layer interacted with amorphous and crystalline Si of two different orientations ((100) and (110)). Interfacial reactions between Co and Si of different type have been investigated at 300 °C. The results were compared with bulk diffusion couples as well.

TRANSMISSION ELECTRON MICROSCOPY STUDIES OF NANOSTRUCTURED TiO\textsubscript{2} FILMS ON DIFFERENT SUBSTRATES

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A chemical vapour deposition (CVD) synthetic route to nanocrystalline titanium dioxide has been carefully investigated on different substrates. CVD was performed at low temperatures of 320 °C onto KCl crystal, Al foil, KBr pellet and freshly sliced mica. Films of approximately 1 µm thickness were obtained from 1M solution of TiCl\textsubscript{4} in dichloromethane (Aldrich). Influence of the substrate to the crystallographic orientation and morphology of the films, regarding the substrate was studied by analytical electron microscopy techniques including bright-field (BF), dark-field (DF), selected area electron diffraction (SAED), high-resolution transmission electron microscopy (HRTEM) and energy dispersive spectroscopy (EDS). Investigations were carried out by using a JEOL JEM 2010 200 kV and a Morgagni FP 5005 microscope. Bright-field (BF) images of TiO\textsubscript{2} samples prepared on different substrates obtained by Morgagni microscope have shown a large number of grains which are differently oriented regarding incoming electron beam and indicated different morphology of each sample. The difference in morphology regarding different substrate was confirmed by JEOL microscope. The average grain sizes were determined from DF images of HRTEM. Table I is showing that different substrates influenced average grain sizes of nanostructured films. The average grain sizes are increasing as the substrate changed from KCl to Al to KBr and finally to mica. The nano-grains in TiO\textsubscript{2} films deposited onto KCl crystal, KBr pellet and freshly sliced mica are of irregular shape, while they are spherical in the film deposited onto Al foil.
SELF-ORGANIZATION OF NANOPARTICLES IN TiO₂ THIN FILM ON THE GLASS SUBSTRATE

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Nanocrystalline titanium dioxide colloids have been synthesized using a sol-gel technique followed by growth under hydrothermal conditions in a basic environment at temperatures between 190°C and 270°C. Thin films have been made from aqueous suspension of these colloids. Grazing-incidence X-ray scattering diffraction (GIWAXD) analysis showed the films to be primarily the anatase crystal phase. It is in agreement with previous scanning electron microscopy (SEM) results, which had revealed a predominantly rod like particle morphology after growth at lower temperatures. The formation of principally truncated tetragonal or tetrahedral bipyramidal nanocrystallites followed growth at higher temperatures. The rod like particles self-organize into regular cubic arrays with the long axis of the rods aligned perpendicular to the film surface. This self-organization is dependent upon the base used in colloidal synthesis.

In this work we have performed GIWAXD experiment at synchrotron ELETTRA in Trieste, Italy. The two-theta angle was changed from 5 to 35 degrees with parallel change of rocking-angle for 4 degrees. Focused on anatase line at 25 degrees we have followed its behaviour upon change of rocking-angle. In basic "ordered" TiO₂ film intensity of this line has changed and thus showed (101) orientation plane parallel to substrate as dominant. This plane is connected with tetragonal and bipyramidal particles that have primarily (101) surfaces, as observed previously for anatase nanocrystallites. In transparent "acidic" films the preferential (412) plane was dominant.

SURFACE OF SPUTTER-DEPOSITED TUNGSTEN FILMS

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Different phases of tungsten were produced by magnetron sputtering either as a single layer or the multilayers. A stable alpha-W phase (b.c.c. structure) is predominantly obtained at low working gas pressure, metastable beta-W phase (A15 structure) is obtained at increased working gas pressure, and amorphous-like tungsten (a-W) is obtained at even higher pressure. The occurrence of various phases is related to the oxygen content and built-in stresses in the prepared thin films. We varied the pressure during the preparation to produce tungsten films composed of stacked layers of different tungsten phases. The surface roughness/ topography of such films was examined by the AFM, and subsurface layer structure by the SAXS and X-ray reflectivity measurements.
Tungsten multilayers were prepared by sequential deposition of tungsten sputtered under different conditions in a cylindrical magnetron device. Working gas pressure was varied in the range of 0.7 – 3.5 Pa, and circular substrates (monocrystalline silicon, borosilicate glass) were cooled in contact with the LN2 container. The deposition rate was about 0.17 nm/s, and the total film thickness range was 50-200 nm.

AFM study revealed complex dependence of surface features on the preparation conditions. SAXS data show presence of surface/ subsurface nanoparticles whose size depends on the top layer phase. Finally, the X-ray reflectivity suggests that the surface roughness is increasing with overall thickness, with Bragg peaks missing in most of the multilayered samples.

**PATTERN FORMATION IN SiSb SYSTEM**


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The thermal annealing of Si/SiSb/Si amorphous thin film tri-layer samples results in an interesting pattern formation. In pictures, taken by means of cross-sectional transmission electron microscopy (TEM), a two-phase, fine-grained structure can be observed, additionally, on much larger length-scale a stripe-shaped contrast, parallel with the substrate and terminal surface of the sample can be seen.

In our previous study we proposed two opportunities to explain the experimental results: a) surface-driven spinodal decomposition; b) crystallization and segregation. Our recent experiments proved that the fine-grained structure remained amorphous, crystallization could not be observed. TEM investigation revealed that generally 3 Sb-rich stripes are formed, one is in the middle of the alloyed layer and 2 stripes appear symmetrically in the outside Si-layers.

In the present work we investigate in details the impact of the layer thickness and the alloyed layer composition on the stripe-shaped pattern formation.

**IN-SITU SPECTROSCOPIC ELLIPSOMETRY OF DLC BY BIPOLAR PULSED DC PACVD**

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Diamond-like carbon (DLC) hard coatings are well known for possessing a number of desirable properties for engineering applications such as decreasing wear and friction. The detailed study of the composition at the very initial stages of growth of these layers has not yet been addressed due to a lack of on-line monitoring techniques. In this contribution DLC films were deposited in a commercially available reactor designed for production of hard coatings as well as for nitriding
by bipolar pulsed DC PACVD. CH$_4$ and H$_2$ were utilized as precursor gases for the DLC films. In-situ real-time ellipsometry was used to monitor the entire deposition process, including heating up of the substrate, the evolution of the film growth itself during the deposition step, and the final cooling down stage of the DLC coating. The measurements have been carried out with a fast spectroscopic ellipsometer recording in the wavelength range from 350 to 850 nm.

**OPTICAL CHARACTERIZATION OF TiO$_2$ AND ZrO$_2$ THIN FILMS BY THE COMBINED METHOD OF SPECTROSCOPIC ELLIPSOMETRY AND SPECTROSCOPIC PHOTOMETRY**

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Thin films of ZrO$_2$ and TiO$_2$ are frequently employed for fabrication of many optical devices in optics industry. However, their optical properties are strongly dependent on technological procedures used to create them. Therefore it is necessary to have reliable optical methods for their characterization. In this contribution we will present the method very efficient and reliable for this purpose. This method is based on interpreting the experimental data obtained using variable angle spectroscopic ellipsometry (VASE) and photometry based on measuring the reflectance and transmittance. The values of the reflectance were measured from both the sides of the characterized films since these films were deposited by RF magnetron sputtering onto the substrates formed by the plan-parallel plates of K64 glass (i.e. from the air side and glass substrate side). All the spectroellipsometric and spectrophotometric data were treated simultaneously by the least-squares method (LSM) for each of the film analyzed. In formulae employed in the LSM roughness of the upper boundaries of the films was respected using Rayleigh-Rice theory (this roughness was accomplished by AFM measurements). The values of the optical constants of the glass substrates were determined independently by the same method using the K64 glass plates free of the films. In the contribution the values of thickness and spectral dependences of the optical constants for the selected films of TiO$_2$ and ZrO$_2$ will be presented as the results of their optical characterization using the method presented.
MORPHOLOGY OF PHOSPHORUS-IMPLANTED p-TYPE SILICON ELECTROCHEMICALLY ETCHED IN HF ELECTROLYTE

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The influence of the initial Si surface state on pore formation has been studied. For this purpose, samples were implanted by phosphorus ions at different doses and energies. Electrochemical characterizations were used on phosphorus implanted p-Si during the anodic polarization, with the SEM observation and the RBS/Channeling technique.

After a short anodization period (5s) in HF electrolyte, the topography of the surface exhibits a high density of pyramidal structures, while the adjacent virgin silicon remains undisturbed. The increase of the polarization time shows the formation of a large square groove at the base of pyramide. The results show that pore grows anisotropically and porous Si growth can be initiated preferentially at surface defects, created in a p-type Si substrate by phosphorus ion beam bombardment.

In practice, they indicate that an implantation of phosphorus ions followed by an electrochemical etching in HF electrolyte can be used both for microstructuring the silicon and/or to form visible light emitting porous Si selectively.

BLUE LUMINESCENCE FROM POROUS LAYERS PRODUCED BY METAL-ASSISTED CHEMICAL ETCHING ON LOW DOPED SILICON

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Photoluminescent porous layers were formed on highly resistive p-type silicon by metal-assisted chemical etching method using K₂Cr₂O₇ as an oxidizing agent. A thin layer of Ag is deposited on the (100) Si surface prior to immersion in a solution of HF and K₂Cr₂O₇. The morphology of porous silicon (PS) layer formed by this method as a function of etching time were investigated by scanning electron microscopy (SEM). It shows that the surface is porous and the layer thickness increases with etching time and is not limited by an instability as observed with electrochemical method. The porous layers was characterized by backscattering spectrometry (BS) as a function of etching in random and channelling mode Channelling spectra show that the pore walls remain crystalline after etching. On the other hand, Random and channelling spectra show that the deposited silver diffuse into the pore. Luminescence from metal-assisted chemically etched layers was measured by photoluminescence (PL) using a He-Cd laser for excitation. It was found that PL intensity increases with increasing etching time. This behaviour is attributed to increase of the silicon nanostructures density. Finally, the PL spectra show two peaks of emission at 450 and 600 nm.
MOCVD GROWTH OF GALLIUM OXIDE THIN FILMS AND THE THERMAL ANNEALING

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Gallium oxide, with a wide band gap of 4.9 eV, has recently attracted interests as a new material for gas sensor, transparent conductor, phosphor, and solar cells. Various techniques have been studied for the production of gallium oxide thin films but the metal organic chemical vapor deposition (MOCVD) has an advantage of good step coverage, uniformity and reproducibility. We have demonstrated the deposition of gallium oxide thin films by MOCVD using the trimethylgallium (TMGa) as a precursor in the presence of oxygen. The XRD data showed that as-deposited gallium oxide films were fully amorphous but very small crystallites of monoclinic \( \beta\)-Ga\(_2\)O\(_3\) appeared by the thermal annealing at a sufficiently high temperature. The AFM analysis revealed that the surface roughness increased by the thermal annealing. Photoluminescence (PL) measurements indicated that both as-prepared and annealed gallium oxide thin films under excitation at 250 nm showed two emission bands at 364 and 466 nm, corresponding to the ultraviolet (UV) and blue region, respectively, but the relative peak intensities of UV and blue emission changed by the thermal annealing. The production of gallium oxide thin films using the conventional source and the possibility of strong emission will shed light on the potential application of gallium oxide films to the optoelectronic devices.

NEW RESULTS OF LOW TEMPERATURE PECVD AMORPHOUS SILICON CARBIDE

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The application of SiC in semiconductor device technology requires controlled and selective doping. Standard technologies for silicon device production, such as ion implantation at room temperature and the subsequent thermal annealing of radiation damage at moderate temperatures cannot be adopted because the radiation damage in SiC is extremely stable. Very high temperatures are necessary for its annealing and to activate dopants. One way to overcome this problem is to perform high current pulse electron or ion beam irradiation instead of high temperature annealing.

Nitrogen-doped amorphous silicon carbide films were grown by a plasma enhanced chemical vapour deposition (PE CVD) technique. Samples with different amounts of N were achieved by a small addition of ammonia NH\(_3\) into the gas mixture of silane SiH\(_4\) and methane CH\(_4\), which were directly introduced into the reaction chamber. The actual amount of nitrogen in the SiC films was determined by Rutherford backscattering spectrometry (RBS). The hydrogen
concentration was determined by an elastic recoil detection (ERD) method. For irradiation experiments we used electron beams with a kinetic energy 200 keV, a pulse duration of 300 ns, and a beam current of 150 A/cm². The number of pulses was altered from 10 to 20 pulses. A simulation of the RBS spectra was used to calculate the concentration of carbon, silicon and nitrogen. It was found that with increased nitrogen doping and following activation of dopants the resistivity of the amorphous SiC films was substantially reduced.

ESTIMATION OF AMORPHOUS SILICON THIN FILMS DENSITY BY OPTICAL METHODS

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Due to leak of long range ordering, density of amorphous silicon is lower than in its crystalline form. By hydrogenation of material, the density difference becomes larger. In order to study the effect qualitatively, the series of thin amorphous hydrogenated silicon films with large variation in hydrogen to silicon ratio, deposited by magnetron sputtering, was examined by UV-visible-IR spectroscopy and nuclear methods.

The film density of deposited films was estimated by using tree different approaches. In the first one, the density was estimated by using EMA (Effective Medium Approximation) analysis of long wavelength dielectric function. In particularly, the basic and modified Bruggeman and Maxwell-Garnett models were tested. The second set of values for density was obtained by analysis of stretching vibrations of Si-H bonds in IR part of spectrum, by using earlier published method. These results were compared with the results obtained by nuclear methods, allowing the discussion of compatibility and accuracy of all of applied methods.

The final result, density variation in a-Si:H samples deposited by magnetron sputtering as a function of hydrogen to silicon ratio, was compared with results of density measurements made on the samples deposited by other techniques. The qualitative and quantitative matching allows some concluding remarks upon intrinsic structural property of amorphous Si:H material.

NiO-BASED THIN FILMS WITH Pt SURFACE MODIFICATIONS FOR GAS SENSORS

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Nickel oxide, which belongs to metal oxides, is usually taken as a model for p-type materials, is an attractive material well known for its chemical stability as well as for its excellent optical and electrical properties. Indeed, NiO thin films have been studied for applications in electrochromic
devices and also as functional layers for solar cells. In particular, the field of gas-sensing has benefited from the production of prospective materials characterized by a high surface-to-volume ratio. The gas-sensing properties of metal oxides are more or less related to the material surface, its high porosity and a microstructure with a small particle. Also, these properties can be essentially improved by doping of their surfaces by catalytic metals.

In our study, the surface modification of nickel oxide thin films using Pt was investigated. Very thin Pt very thin overlayers with a thickness of about 3 and 5 nm have been deposited on the top of the NiO surface by magnetron sputtering. Thus, the modified NiO films have been investigated respect to surface microstructure and morphology and hydrogen sensing properties. The Pt-modified NiO sensors showed high responses and sensitivities to very low concentration (500÷5000 ppm) of H₂ in air; by taking into account that the lower explosion limit (LEL) range of H₂ is 40,000 ppm. The sensor response to H₂ resulted to be strongly dependent not only on the grain size of Pt cluster islands but also on the porosity of the Pt film on the NiO surface. These results make the Pt-modified NiO sensors promising candidates for implementing hydrogen leak detector.

**PLASMA CVD OF ALUMINA – UNSOLVED PROBLEMS**

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Due to their excellent chemical stability in combination with advantageous mechanical properties, hard alumina (Al₂O₃) coatings have attracted quite some interest in the tool industry. However, such coatings are usually produced by chemical vapour deposition (CVD) at temperatures above 1000 °C. Hence, these coatings were applicable only for tools made of cemented carbide. Finding a process, which would allow hard alumina deposition at significantly lower temperatures would be rather desirable.

In the present investigation a commercially available plasma CVD system suited for the deposition of titanium nitride (TiN) was adapted for alumina deposition up to substrate temperatures of 800 °C. Besides of a modification of the gas supply system, an additional heating system inserted into the reactor was needed. Bipolar voltage pulses were applied to the substrate holder whereas the grounded reactor wall served as counter electrode. To achieve a uniform discharge across the substrate holder an additional grounded counter electrode was placed close to the substrate holder.

The investigations clearly demonstrated that activating the process gas by plasma lowers the temperature needed for the formation of hard alumina coatings. The properties of the alumina coatings depend strongly on process parameters like temperature, plasma power and process gas composition. Higher plasma power as well as certain additives to the process gas favour the formation of hard alumina at lower temperatures and suppress the formation of softer alumina modifications. At the present stage the process is still not sufficiently uniform. Furthermore, certain types of damage in the coatings were observed.
**MORPHOLOGY OF COMPOSITE FILMS WITH NON-SPHERICAL OBJECTS**

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Composite films consisting of metal particles or even nanoparticles embedded into an oxide or polymer matrix have received more and more attention in last few years due to their interesting optical, mechanical, and electrical properties. Knowledge about the structure of composite films of various types is crucial for the appreciation of what properties the structure would have. Most modern materials including composites typically have a complex three-dimensional structure on either a micrometer or nanometer-scale. However, while most microscopic techniques currently available are limited to a two-dimensional representation, a detailed study of the relationship between material structures and properties is restricted thereof. The situation provides an opportunity for the application of methods of computational physics.

The paper deals with the possible statistical reconstruction of the three-dimensional structure when only one two-dimensional image and some additional information are available. The survey of methods convenient for reconstruction of size and/or spatial distribution of objects of which the composite structure consists is done. The models of the composite films uses usually symmetrically formed particles like spheres. However, we wanted to find out the possibility of reconstruction in not so easy cases when the particles in the film have more complex shape. We have prepared models of composite films with the ellipsoidal or polyhedral forms of objects and studied the unfolding problems arising from the image analysis of the film.

**WEAR AND FAILURE MODES OF PVD COATED SOLID CARBIDE SLITTING SAWs**

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Wear and failure modes of PVD coated solid carbide (HM) slitting saws, which are used for large scale production of small commutators, have been studied by scanning electron microscope (SEM), atomic force microscope (AFM) and profilometer. Such saws are used for cutting the commutator body, which is composed of two parts: the outer ring made of electrolytic copper, and the inner ring made of hard plastic (bakelite filled with glass fibers). Strong wear of the rake face of cutting teeth as well as of the cutting edge were observed. We have improved the performance of HM saws significantly by deposition of various PVD hard coatings. In this work we will present the results of SEM, AFM and profilometer analysis of HM saw teeth, which performed different numbers of cutting cycles.
THERMALLY STIMULATED CURRENTS MEASUREMENTS ON SEMI-INSULATING GaN THIN FILMS

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Thermally Stimulated Currents Measurements (TSC) was used in the study of deep levels in semi-insulating GaN thin film samples, grown on sapphire by the molecular beam epitaxy. Prior to the standard TSC measurements samples were subjected to "thermal cleaning procedure" at certain temperatures in 98 – 250 K range. So obtained TSC spectra were all successfully fitted by the Simultaneous multiple peak analysis method (SIMPA), using the same set of deep levels with activation energies between 0.09 and 0.60 eV, differing only in relative concentrations.

MAGNETRON SPUTTERING FABRICATION OF ERBIUM DOPED GALLIUM NITRIDE THIN FILMS

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Gallium nitride (GaN) is a promising material for optoelectronic applications. Erbium containing GaN can be used as basic part of solid state lasers or optical amplifiers operating at 1 550 nm.

The GaN films were deposited by RF magnetron sputtering (Balzers Pfeiffer PLS 160). Two basic approaches of the RF magnetron sputtering of the GaN thin films in nitrogen-argon gas mixture on different substrates at room temperature are presented. The first one, utilising the metallic gallium target, provides deposition of the well-developed polycrystalline layers. The second one, with using gallium oxide target, resulted in almost amorphous GaN films with poorly developed crystals. Both approaches allowed doping the GaN films with erbium ions during the deposition. For that, the Er metallic powder was laid on the top of the gallium target, or, Er₂O₃ pellets were put on the top of the Ga₂O₃ target.

The amount of the incorporated erbium depended on the area of the target covered by the erbium powder as well as on the erosion area represented by the part of the surface covered by the Er powder or Er₂O₃ pellets. The reasons for better 1 550 nm luminescence properties of the latter are under investigations.

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MERCURY-PLATED THIN FILM ITO MICROELECTRODE FOR STRIPPING ANALYSIS OF TRACE HEAVY METALS

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Anodic stripping voltammetry (ASV) with mercury plated metal thin electrodes (Ir, Pt, Au, Ag) is one of the most sensitive and widespread method for determination of trace heavy metals (Cu\textsuperscript{2+}, Cd\textsuperscript{2+}, Pb\textsuperscript{2+}, Zn\textsuperscript{2+}) in environmental analytes.

We have developed a mercury-plated array of planar microelectrodes based on Indium Tin Oxide (ITO) thin film, which was manufactured by thin-film technology on a silicon substrate. Advantages ITO films are in their high electrical conductivity (\(> 10^5 \, \Omega^{-1} \, \text{m}^{-1}\)), optical transparency (\(> 80\%\)), and simplicity of chemical patterning. Two microelectrode arrays containing 100 microdiscs each with a diameter 20 \(\mu\text{m}\) or 40 \(\mu\text{m}\), respectively, and interelectrode distances of 200 \(\mu\text{m}\). The ITO films (600 nm) were prepared by rf reactive sputtering (Ar + 5\% O\(_2\)) from In\(_2\)O\(_3\)-9mol\% SnO\(_2\) target of purity 99\% on Si/SiO\(_2\) substrate externally heated to temperature of 350 \(^\circ\text{C}\). An electroplating of mercury onto the ITO microelectrode was carried out in a solution of 1 mM Hg(NO\(_3\))\(_2\) and 0.1M KNO\(_3\) by applying potential –1.5V vs. Ag/AgCl/Cl\(_2\) for varying time. The surfaces of modified electrodes were analyzed by optical and atomic force microscopy (AFM). The behavior of ITO array of microdisc electrodes has been characterized by cyclic voltammetry.

In \textit{ex-situ} detection of Pb\textsuperscript{2+} in aqueous solutions by ASV with mercury plated ITO microelectrodes were obtained different current responses depending on the mercury plating conditions (deposition potential and time, concentration of mercury plating solution) and preconcentration step conditions (preconcentration potential and time).

TEM STUDY OF THE MICROSTRUCTURE OF CdSe PREPARED BY SUCCESSIVE VACUUM DEPOSITION OF THIN Cd LAYERS AND Se

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CdSe is a 1.7 eV band gap semiconductor with photovoltaic effect, providing applications in solar cells, transistors, photoconductors, gas sensors and photoluminescent devices.

CdSe thin films were prepared by successive vacuum deposition of Cd and Se. 30-60nm Cd was deposited onto 1.5 nm thick Ag seed-layers on (001) (011) and (111) NaCl pre-treated with Cl\(_2\) [1] and on carbon foil. 5-120 nm Se was deposited at 0.01-0.1 nm/s onto the Cd layers at 100-200 \(^\circ\text{C}\). The Cd-Se reaction occurred during the Se deposition [2].

TEM revealed (0001) Cd with two possible azimuths on (001) and (011), one azimuth on (111) NaCl and random orientation on carbon foil.
SAED of partly selenized samples showed CdSe[10.0]/Cd[10.0] orientation indicating topotactic Cd-Se reaction. The layers exhibited small grains with crystal defects and high stress field. Appearance of Kirkendall voids represented diffusion transport of Cd.

As the rest of the Cd was consumed, a re-crystallization of the CdSe occurred associated with grain coarsening and a re-orientation. This is attributed to the transfer from a two-phase (Cd/CdSe) to a one-phase (CdSe) system and the influence of the free surface energy of CdSe instead of the interface energy of Cd/CdSe. Fully selenized CdSe layers were stoichiometric (50/50 at%) measured by EDS.

The results may suggest alternative technology for the preparation of thin films and devices of II-VI semiconductors.

SiGe-based binary alloys have generated a lot of interest both fundamentally and technologically and have emerged as the materials for photo voltaic, opto electronics as well as other applications. By alloying hydrogenated amorphous Si with Ge, it is possible to decrease the band gap and thus improving the long wavelength response of the solar cells. The improvement in opto-electronic properties of a-SiGe:H alloys and also the solar cell structure have also been attempted. The deposition of thin films for solar cell structure on the low cost and the flexible substrate like plastic foil have necessitated the deposition of the thin films at relatively low temperature. Although many studies have been undertaken on the hydrogenated amorphous silicon germanium alloys, very few studies have been made on rf sputtered a-SiGe:H films deposited at relatively low temperature even though the rf sputtering is an efficient process to prepare thin films of amorphous semiconductors and metals. In the present manuscript we have studied the rf sputtered hydrogenated amorphous silicon germanium alloy films deposited at room temperature onto various substrates including plastic foil and have discussed their mechanical and electronic properties for the possible application in thin film solar cells.

Hydrogenated amorphous SiGe alloy films were prepared using reactive sputtering technique by sputtering simultaneously the high purity silicon and germanium targets in a modified high vacuum sputtering apparatus (Leybold Z400). More than hundred samples with good reproducibility have been deposited for different measurements. The films of different germanium composition were deposited on to various substrate with different hydrogen concentrations. Some samples were also annealed at 300 °C for an hour in the hydrogen atmosphere after the growth process.

To determine the Ge content of the studied film, RBS measurements using He beam was performed in a scattering chamber with a two-axis goniometer connected to a 5 MeV Van de Graaff accelerator. Further properties by optical methods such as ellipsometry are investigated.

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prepared from the mixture of hexamethyldisiloxane (HMDSO) or hexamethyldisilazane (HMDSZ) with nitrogen using plasma enhanced chemical vapour deposition (PECVD) techniques. The hydrogen and/or nitrogen evolved from polymeric films were studied by thermal desorption spectroscopy (TDS) with constant heating rate. The bonding of hydrogen and nitrogen in samples was investigated by IR absorption measurements of samples in as-deposited and annealed state.

CHARACTERIZATION OF ZnO LAYERS PREPARED BY PULSED LASER DEPOSITION

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The new material technology and preparation such of ZnO contains new methods like the pulsed laser deposition. The pulsed laser deposition allows us to prepare the ZnO with two different methods. One of them is the Zn target sputtering in O2 atmosphere and the second one is the preparation directly from ZnO target sputtering in O2 atmosphere onto a sapphire and Si substrates with a high power pulsed laser. The characterizations were made by atomic force microscopy to ensure the layer thickness, roughness, homogeneity. Other chemical qualification was done with the secondary ion mass spectroscopy and EDX in the order investigate the composition and the impurities of the atomic layers. The investigation of surface morphology was performed by SEM.

STRUCTURAL AND OPTICAL PROPERTIES OF ZINC OXIDE FILMS ON PLATINUM OR GOLD-COATED SUBSTRATES

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Zinc oxide (ZnO), a semiconducting, photoconductive, piezoelectric, and optical wave material, shows a wide range of scientific and technological applications. We have demonstrated the deposition of ZnO thin films on platinum (Pt) or gold (Au)-coated substrates using the diethylzinc (DEZn) as a precursor in the presence of oxygen. To our knowledge, this is the first report on the preparation of ZnO film on metallic substrates using the MOCVD. XRD data showed the strong (002) peak, indicating that the films were grown with a c-axis orientation. SEM images also revealed that the ZnO film structure consisted of some columnar-structured grains, representing c-axis oriented grains. Photoluminescence (PL) spectra under excitation at 325 nm revealed that ZnO films on Pt and Au substrates, respectively, showed broad and strong emission bands with peaks at 380 nm in ultraviolet region and at 430 nm in blue region, respectively, indicating the dependency of the peak position on the substrate material. Furthermore, the overall intensity of the emission band was stronger when the Au substrate was
employed. We have discussed the possible mechanism which makes differences in the optical properties of ZnO films.

CHARACTERIZATION OF TITANIUM SILICIDE THIN FILMS

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We report on properties of titanium silicide thin layers prepared on P-doped (100) Si substrates by sputtering of Ti layer followed by a rapid thermal annealing (RTA) in nitrogen atmosphere at different conditions. Thicknesses of as-deposited Ti layers were in the range of 40±60 nm, the annealing temperatures were in the range of 750±900 °C. Formation of a compact TiN/TiSi₂/Si structure by RTA has been revealed by Auger Electron Spectroscopy combined with ion beam sputtering, and confirmed by Scanning Electron Microscopy (SEM) / EDS X-ray microanalysis of mechanically beveled samples (bevel angle of 0.2 degree). Presence of both carbon and oxygen of a different content in TiN layers has also been detected. Both C₅₄ and C₄₉ TiSi₂ phases were identified by micro-Raman spectroscopy in samples annealed at 750 °C, whereas mainly C₅₄ phase has been found in samples annealed at higher temperatures. Creation of isolated TiSi₂ grains of a deep sub-micrometer size at TiSi₂/Si substrate has been found out by the SE mode of the SEM. These grains are gradually merging into homogeneous but grainy TiSi₂ layer. The roughness mean value of the resulting TiN/TiSi₂/Si structures, measured by Atomic Force Microscopy, has been in the range of 24±50 nm. This confirms the grainy nature of prepared TiN/TiSi₂/Si structures.

The work has been carried out in the frame of the Ministry of Education project VTP AV/805/2002 and supported by the Slovak Grant Agency grants 1/9042/02, 1/0152/03, and 1/1054/04.

NITRIDED COATING ON THE SURFACES OF THE γ-TIAL ALLOYS

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Nitrided coating on the surfaces of Ti-47Al-2Nb-2Cr and Ti-47Al-2Nb-2Mn-0.8TiB₂ was done in 10 cm⁻³.s⁻¹ NH₃ at 1,000-1,300 K. The values of mass change of the alloys were fitted to the Arrhenius type equation and the activation energies for the mass change were calculated. Knoop hardness of the alloys were successfully improved. The nitridation depth of the alloys was determined using Rutherford Backscattering (RBS) and the elemental line scanning (EDX)
techniques. The activation energies of the nitridation depth from the two techniques were determined and compared to that of the mass change results. In addition, the two alloys were analysed using an x-ray diffraction (XRD) to determine the formation of the new phase(s).

**Keywords:** Nitridation, γ-TiAl, RBS, EDX, XRD

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**DETERMINATION OF THE STATE OF THE PIGMENT DISPERSION IN POWDER COATINGS**

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The state of the pigment dispersion in powder coatings is described by the pigment size distribution and the average particle size. Quantitative results were obtained using selective oxygen plasma etching, scanning electron micrography and image analysis.

In order to correlate the differences in the production equipment, two sets of experiments were made with the same formulation of the powder coating; one, using three different extrusion units, and the second, with the same equipment but with different input power. The results are compared to those obtained using colour difference measurements. Pigment size distribution plots correlate well with measured colour differences.

**INFLUENCE OF TEMPERATURE ON GROWTH OF AEROSOL PARTICLES BY CHEMICAL DEPOSITION**

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A number of processes taking place in the modern technology and in the atmosphere are related to chemical deposition from the gas phase onto aerosol particles (e.g. manufacture of nanoparticles, chemisorption of molecules on aerosol particles, formation of smog particles). The paper deals with the theoretical study of the growth of aerosol particles by chemical deposition. Dependence of the particle growth rate on the temperature is investigated. Equation for particle growth rate in chemical deposition giving limiting cases in relation to Knudsen number and taking into account adsorption of foreign (buffer or admixture) gases on the particle surface has been obtained. It is shown that the particle growth by chemical deposition depends
on the ratio of the desorption energy of the reactant molecules to the activation energy of heterogeneous chemical reaction and that the growth rate of the particle as a function of the temperature can have a maximum. Effect of the temperature on the composition of the particle growing by chemical deposition from a gas mixture is discussed.

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TRIBOLOGICAL CHARACTERISTICS OF CrCN COATINGS AT ELEVATED TEMPERATURES

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The tribological behaviour of CrCN coatings prepared by unbalanced magnetron sputtering were studied in this paper. CrCN coatings have not been intensively studied yet although some special tribological properties could be achieved. The investigations on the wear performance of CrCN film through wear testing under identical conditions demonstrated that this film possesses better wear resistance than TiN. Nevertheless, the knowledge of wear properties of CrCN coating at elevated temperatures has been still very limited. To help remedy this lack, they were studied with respect to their friction and wear properties at temperatures in the range from RT to 800 °C.

The substrates made from austenitic steel were coated with CrCN coatings during one deposition. The measurements were provided by high temperature tribometer (ball-on-disc, CSM Instruments) allowing to measure the dependency of friction coefficient on cycles (sliding distance) at the temperature up to 800 °C. The evolution of the friction coefficient with the cycles were measured under different conditions, such as temperature and sliding speed, and the wear rate of the ball and coating were evaluated. As counter-parts the balls 100Cr6, Si3N4 and Al2O3 were used. The wear tracks were examined by optical methods and by SEM. The surface oxidation at elevated temperatures and profile elements composition of the wear track were also measured.

THERMAL STABILITY OF THE PLASMA DEPOSITED DIAMOND-LIKE CARBON FILMS

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The thermal stability of diamond-like protective (DLC) films plays a crucial role for their technological application and its study is of great importance. The aim of this contribution is to present the results achieved in our studies concerning the thermal stability of the optical and mechanical properties of DLC films prepared under several different deposition conditions onto
silicon single crystal substrates. The effect of silicon and oxygen incorporation on the thermo-mechanical stability of the amorphous carbon network were investigated.

The optical properties of the DLC films were studied using the method based on the simultaneous treatment of the experimental data obtained by variable angle spectroscopic ellipsometry and spectroscopic reflectometry within the spectral region 190-1000 nm. Using a new dispersion model based on parameterization of the density of electronic states the dependences of the material parameters and the ratio of sp$^3$ and sp$^2$ carbon bonding configurations of the DLC films on annealing temperature were determined.

The mechanical properties were studied by depth sensing indentation test. We found good correlation between the optical and mechanical properties. We observed the decrease in hardness, in fracture toughness and in sp$^3$/sp$^2$ ratio due to annealing. The behavior of the hydrogen in the films was investigated using thermal desorption spectroscopy.

THE INFLUENCE OF DEPOSITION PARAMETERS ON TiB$_2$ THIN FILMS PREPARED BY DC MAGNETRON SPUTTERING


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TiB$_2$ thin films were deposited on Si (001) substrates by DC magnetron sputtering from the stoichiometric target in the Ar inert atmosphere. The dependence of microhardness and adhesion of the coatings on argon pressure and substrate voltage bias was investigated. The properties of the coatings by AES, XRD, AFM, microindentor and scratch tester were examined. Ti/B ratio of the coatings was ½. The coatings morphology and preferred orientation were pressure dependent. Well-adhesive coatings acquired the microhardness of more than 5000HV$_{0.05}$.

RELAXATION PROCESSES AND ELECTRONIC STRUCTURE OF As$_2$S$_3$ THIN FILMS

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In this work time stability in the range from 10 to $10^8$ s along with thermal and laser treatment effect on the following structure sensitive film parameters: stress, refractive index and absorption edge, for 1 µm thick As$_2$S$_3$ thin films deposited by thermal evaporation in vacuum at a pressure of $10^4$ Pa have been studied. The peculiarities in the behaviour of the investigated parameters under the indoor storage of the films have been observed for the times of 10 s, $2 \times 10^2$ s and $2 \times 10^5$ s. It has been found that the shift of absorption edge and the change in
refractive index (n) of the films depend on the value and sign of stress (σ). For the compressive stress we have found \( \frac{\partial n}{\partial \sigma} = 2 \times 10^{-9} \text{ Pa}^{-1} \) and for the tensile ones \( \frac{\partial n}{\partial \sigma} = 0.43 \times 10^{-9} \text{ Pa}^{-1} \). After annealing the tensile stress in the films and the refractive index tend to increase, and \( \frac{\partial n}{\partial \sigma} = -0.33 \times 10^{-9} \text{ Pa}^{-1} \).

The calculation of the top of the valence band, optical band gap, position of the energy levels formed in the band gap by homopolar bonds and clusters have been performed by the linear combination of atomic orbitals method in the context of V₁-bands model. Primal and inverse problems for electronic structure simulation have been solved accounting experimental photoemission and photoluminescence spectra together with mechanical and optical properties.

### DIAMOND-LIKE CARBON THIN FILMS FOR HIGH TEMPERATURE APPLICATIONS PREPARED BY FILTERED PULSED LASER DEPOSITION

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Diamond-like carbon thin films (DLC) with increased thermal stability were prepared by filtered pulsed laser deposition (F-PLD). The influence of different substrate temperatures on the growth and composition of DLC films was analysed using Raman spectroscopy and electron energy loss spectroscopy (EELS). The increase of substrate temperature leads to an increase in sp² carbon bond hybridisation and a decrease in the optical band gap. However, the deposited films show excellent thermal stability, with a slow transition from high sp³ to low sp² content as a function of the substrate temperature. The importance of substrate temperature and the mechanical kinetic energy filter in relation to the mobility of the film-forming species from the highly ionised and energetic laser plume is discussed.

Keywords: DLC films; filtered PLD; EELS; Raman spectroscopy; transition temperature; thermal stability

### ELECTRICAL AND OPTICAL PROPERTIES OF SnO₂:Sb THIN FILMS PREPARED BY CVD METHOD

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This work outlines the fabrication and characterisation of transparent doped and undoped tin oxide layers deposited by chemical vapour deposition (CVD). The thin films were grown on glass substrates at atmospheric pressure and temperature varying between 350°C and 420°C in
an horizontal furnace. The growth rates of 140 Å/min have been achieved for substrate temperature and flowing oxygen of 400°C and 1l/min, respectively. The as-prepared films are of polycrystalline structure, with 200 preferential orientation. Examination of the surface morphology by scanning electron microscope (SEM) showed that the SnO₂ thin films are textured, made up of many pyramidal grains with average grain size of 200nm. Electrical resistivity measurements were performed for dopants concentration (Sb/Sn) varying from 4% to 0.66%. The most conductive films were obtained for antimony dopant concentration of 0.88% with resistivity of 6·10⁻⁴ Ωcm, the higher optical transmission, about 75% and 85% was obtained for antimony dopant concentration of 0.66% and undoped samples, respectively.

MORPHOLOGY OF NANO-SIZED GADOLINIUM PARTICLES PREPARED BY GAS-DEPOSITION METHOD: ANNEALING EFFECTS

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Gadolinium is the rare-earth material promising for applications in electronics. Gd nanoparticles, in comparison with conventional material, could possess outstanding optical and magnetic properties. Thermal deposition in inert gas atmosphere is very simple and effective method for preparation of nanoparticles, whose properties can be controlled by variations in deposition and further treatment conditions.

Thin films of Gd were prepared by thermal deposition of Gd in He atmosphere with pressure of several Pa.

The deposited films were characterized by specular X-Ray reflectivity (SX), AFM and In-plane XRD measurements. SXR studies showed that the the deposition rate depends on the pressure in the chamber and the amount of Gd in evaporation cell. The interdiffusion of Si and Gd takes important role in formation of Gd/Si interface and results in creation of GdSi layer of about 3 nm thickness.

XRD measurements showed that Gd nanoparticles grow and partially crystallise after annealing to 600–800°C. The diffraction pattern confirms the presence of partially oxidised Gd cubic phase with lower limit of crystallite size of about 7 nm.

Growing of particles after annealing was observed by AFM. It was found that the particle size depends on deposition and annealing conditions.
IR/UV THIN FILM OPTICAL FILTER FOR AUTOMATIC LCD WELDING FILTER

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Automatic LCD optical filters are becoming increasingly important as personal protective devices. As their primary task is the protection of human eye, very strict and demanding requirements apply to their technical performances. The main active optical element, LCD light shutter however cannot possibly comply with all safety requirements in UV and IR light spectrum and LCD light shutters have to be complemented with thin-film optical filters which extend the safety performances into the UV and IR range.

The paper presents the design of a specialized thin film filter, transparent in the visible, while highly reflecting light in the IR and UV spectral range. The deposition technology development is described including individual oxide and metal layer deposition parameters, results of structural and surface analyses and other characteristics:

The spectral analysis using Perkin Elmer 900 spectrophotometer support the filter design using the multilayer filter computer modeling software. The overall filter performance evaluation was made by AES, TEM and EDS analyzing techniques. The oxide as well as metal detailed layer surface structure was constantly monitored by AFM microscopy, while the refraction indices of individual layers were measured by ellypsometer. The filter surface was evaluated for possible optical defects by Tencor Alphastep 2000 as well as with Leitz metalloplan optical microscope. Mechanical properties of the filters were evaluated as well, using standard methods.

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DYNAMIC EXPANSION SYSTEM – NEW PRIMARY HIGH VACUUM STANDARD OF CMI (10⁻⁴ – 0.1) PA

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The new primary high vacuum standard of Czech Metrology Institute (CMI) is based on dynamic expansion principle. It was developed in cooperation with Charles’ University in Prague and company VAKUUM PRAHA.

Dynamic (continuous) expansion method is based on the expansion of the gas medium through an orifice with known conductance. Continuous flow of gas to the entrance of the orifice is ensured by a flow meter and after passing it the gas is continuously pumped. The relation of the pressures on both sides of the orifice can be evaluated from the first principles.

The presentation will describe the design and evaluation of the mentioned above instrument. It will be focused first of all on the calculation of conductivity of the vacuum orifice, the low gas flow meter design, the determination of the effective pumping speed and its stability and the reduction ratio. It will show the limits of the apparatus and also detailed determination of its uncertainties.
A HIGH TEMPERATURE DIRECT PROBE FOR MS AND ITS USE FOR THERMAL DECOMPOSITION MONITORING

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A high temperature direct probe has been constructed and coupled to a quadrupole mass spectrometer (QMS – Q300C, ATOMKI). The probe is heated by a Thermocoax SEI 15/50 heating element and controlled with a universal PID controller (Omron E5CK). The probe can be used in the range from room temperature up to 800 °C. Programmable parameters: number of steps in which the ultimate pressure is achieved, rate of heating and the length of constant temperature periods in each step. The replaceable quartz and stainless steel cylindrical sample holders immerse into the axial hole (φ4x12) mm on the heated tip of the probe. Quantity of sample material required for the analysis is in the range of 0.1-10 mg. A 25 mm long thin-walled tube separates the hot tip from the water-cooled body of the probe. From ambient pressure the probe is gated through a ball valve to the ion source of the QMS.

After the probe and the complete system had been tested with salts as CaCO₃ and Ca(COO)₂⋅H₂O, it was used to study the thermal reactions and decomposition of mineral samples. The major phase of the samples was quartz and buddingtonit was suspected as the minor phase. The latter is a feldspar with ammonium content (empirical formula: NH₄AlSi₃O₈⋅0.5H₂O). Selected ion monitoring showed high peaks of ammonia at 80, 390 and 490 °C and loss of water at 30 °C, proving the considerable amount of buddingtonit.

NOVEL MODULAR CONTROLLER FOR QUADRUPOLE MASS SPECTROMETERS

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High performance single quadrupole mass spectrometers nowadays are used in a very wide range of applications. Some examples are accurate residual gas analysis down to the XHV range, vapor beam analysis and control in UHV-MBE tools, isotope ratio measurements, quantitative gas analysis in e.g. catalytic research, and process control in the chemical, pharmaceutical, and metallurgical industry. The very different demands of these applications can be fulfilled by very flexible and modular mass spectrometer controllers only. A novel system contained in a only 3u high 19 inch rack system has been developed that shows the following key features: Intel X-scale processor based controller with Windows CE.net operation system; high speed digital signal processor for mass spectrometer control and data acquisition to allow for scan rates down to 125 microsec / amu; communication to host computer via USB or Ethernet for network operation using OPC standards; fieldbus module to communicate with standardized industrial input / output modules; webserver for control of the system without the need for specific software; modular ion source supply with digital emission current controller for easy adaptation.
to different filament types, and individually exchangeable voltage supplies for ion lenses; voltages can be biased up to +/-1000V and polarity can be changed to detect positive as well as negative ions; ion counter for count rates up to 130MHz; high voltage supplies for operation various SEMs; autorange control for electrometer amplifiers; analog and digital input / output module for easy connection to external measurement and control devices. In combination with a wide selection of quadrupole rod systems, RF stages, ion sources and ion optics, this system allows for numerous applications from basic research to industry. Detailed functional description as well as data from selected applications will be presented.

DESIGN OF A CONSTANT PRESSURE GAS FLOWMETER FOR THE RANGE $10^{-6}$ – $10^{-3}$ Pa×m$^3$/s USING "DRY" WELDED BELLows

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Basic parts of many primary standards for vacuum gauge calibration in high vacuum range are flowmeters, i.e. devices generating highly accurately known gas throughput. Mainly the constant-pressure-flowmeters (CPF) are designed for these purposes in recent decades especially for their metrological advantages. The crucial part of the constant-pressure flowmeter is the volume-varying part (VVP).

A new CPF for the gas throughput range $5\times10^{-6}$ – $5\times10^{-3}$ Pa×m$^3$/s was designed as a part of orifice flow primary vacuum standard in Common Laboratory of Czech Metrology Institute and Charles University Prague. A "dry" (directly deformed – without liquid load) welded bellows electrically driven and with electronically measured position was used as VVP. Good reproducibility of the positioning, which is crucial for this solution, was proved experimentally. Non-linearity was overcome by means of careful calibration. The flowmeter with this VVP has very high ratio "maximal volume : minimal volume" – comparable as it is at using "dry" piston and it can be designed entirely uhv tight.

CORRECTION OF THE ORIFICE CONDUCTANCE VALUE IN AN ORIFICE FLOW STANDARD AT SLIGHTLY TRANSITIONAL GAS FLOW REGIME

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Orifice flow vacuum standards are often used for pressure range bellow approximately 10-2 or slightly above despite it would be advantageous to extend this upper limit up to the value 1 Pa or
even more towards the lower limit of the piston gauges. Main difficulty is to manufacture and measure accurately such a small orifice of desired geometrical shape because molecular flow is assumed.

The orifice of usual size (molecular conductance some l/s) could be used in a slightly transitional flow. The Knudsen number Kn at 1 Pa and at 10 mm orifice diameter is still greater than 0.5 and a simple correction of the orifice conductance is recommended in this case \( C \approx C_{\text{MOLECULAR}} \times (1 + 1/X \times Kn) \). The factor X is taken 8 based on computation performed in sixties of 20th century.

The factor X was measured by means of the orifice flow standard. The stability of the used pump was checked by means of the flowmeter method for pumping speed measurement. The knowledge of the factor X enables to extend the range of the orifice flow standard and also to check the theoretical results.

**SIMPLE VACCUM EXPERIMENTS FOR UNDERGRADUATE STUDENT LABORATORIES**

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Education and training of researchers and technicians by actual practice in the laboratory has rather importance and significance in a field like vacuum science and technology. This is of particular importance for undergraduate university education. It is very important that direct quantitative measurements can be obtained during experimental classes, rather than just the simple operation of vacuum systems and qualitative analysis.

We will describe simple experiments intended for didactic laboratory vacuum classes of undergraduate courses where actual measurements are performed and compared with the values tabulated in the literature. Small high vacuum systems are used with low vacuum gauges in both high vacuum chamber and forepump inlet. This allows the monitoring of the pressure in the vacuum chamber during the roughing procedure and after the high vacuum valve is closed. This setting enables a straightforward evaluation of the gas throughput that enters the chamber volume, due to the materials inside the chamber or to a real leak, by the pressure-rise rate. The high vacuum pump and the forepump pumping speed, at the high vacuum chamber, the roughing-line conductance and the outgassing rate of different materials placed inside the chamber can be easily determined.

With an RGA students are able to perform direct quantitative measurements of isotopic abundances and of permeability constants of different materials to hydrogen, helium, neon and other elements (e.g. using the pressure-rise method). He-leak detectors can be used to measure the helium permeability constant of different materials and its dependence on material thickness, area and helium pressure differential.
EXPERIENCES WITH NEW CMI PRIMARY STANDARD OF VACUUM DOWN TO 0.1 PA, BASED ON PISTON DESIGN

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The lower limit of vacuum range of the classical rotating piston manometers ends in the order of kilopascals. The primary standards of lower vacuum ranges have until recent times been based on liquid columns or expansion systems only. The utilization of the digital non-rotating piston manometers for this purpose is very promising.

Czech Metrology Institute (CMI) has developed its own primary vacuum standard based on the commercially available instrument DHI FPG 8601. Some improvements of the residual vacuum generation and measurement and resolution of this instrument were performed. Its range in absolute mode is now from 0.1 Pa to 15 kPa.

Its principle is based on the non-rotating piston centred in the cylinder by the means of low gas flow conveyed into the gap between them. The piston is connected via a trapeze to a mass comparator that indicates and compensates even very small changes of the forces acting on it. Thus the pressure above the piston can be defined from the change of force acting on the piston since the zeroing of the instrument and the effective area of the piston-cylinder.

The presentation will show the entire summary both of the theory of this instrument and practical problems of its primary and secondary traceability. The uncertainty budget as well as the experimental results will be included. The experiences with the operation and international comparison will also be shown.

MAGNETRON TYPE SPUTTER ION PUMP FOR UHV SYSTEMS

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A new type of a sputter-ion pump based on a magnetron discharge is described. The pump consists a combination of Penning and magnetron cells. The cells are constructed from stainless steel anodes with the titanium cathode rods placed in magnetron cells. Cathode plates perpendicular to the anode axis are replaced with small discs in Penning cells. The magnetic field of 0.15 T is kept constant. A stable discharge was established in the pressure range between 1·10⁻⁸ mbar and 1·10⁻⁶ mbar. The discharge current versus the anode voltage was investigated. The results showed that the minimal anode voltage should be 5 kV, since at lower voltage the current is too small. The pumping speed for nitrogen was measured at the anode voltage of 6 kV. The maximum pumpingspeed of 20 l/s was reached at the pressure of 6·10⁻⁷ mbar.
CHARACTERISTICS OF MAGNETRON CELLS WITH DIFFERENT CATHODE RODS

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Magnetron cells can be used as a vacuum gauge or as a part of a sputter ion pump. Discharge characteristics of the magnetron cells with different shape of the central electrode are presented. The experimental cells consisted of an anode cylinder made of stainless steel. The length of the anode cylinder was 35 mm and the diameter was 25 mm. On the axis inside the anode cylinder, a titanium cathode rod was placed. The cathode rod had one of the following shapes: circular, rectangular, square, star or triangular. Discharge characteristics of the cells with different rods were measured systematically at different voltage between 1 kV and 8 kV and the pressure range between $1 \cdot 10^{-8}$ and $1 \cdot 10^{-6}$ mbar. The magnetic field density was constant at 0.13 T. Experimental results showed that the ion current at first slowly increased with increasing voltage, reached a maximum at a certain voltage and decreased with further increase of the anode voltage. The highest currents were observed in the cell with triangular cathode rod, while the lowest currents were observed in the cell with circular cathode rod, where the currents were lower for about 50 %.

CRYOSTAT FOR TRANSFORMER MADE OF HIGH-TEMPERATURE SUPERCONDUCTOR

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Invention of materials which exhibit superconductivity at temperatures as high as 100K has stimulated development of electrical engines, generators, and transformers with electromagnets which are superconductive when cooled by liquid nitrogen only. Since the critical currents of the "high-temperature" commercial superconductors are rather low at the normal boiling point of nitrogen, temperatures about 70K are mostly required in technical applications. Additionally, there are specific requirements which cryostats for ac electrical devices exploiting of superconductive windings should fullfil:

- electric conductive materials (metals) can not be used to avoid an excessive heating by the inductive currents,
- thickness of the thermal insulation is strongly limited by the gap between windings cryostat has to be vacuum tight, including seals of demountable joints,
- pumping system has to guarantee the sufficiently low vacuum and to stabilise it at required values.

An arrangement of an cryostat developed the for experimental superconductive transformer that fulfils the requirements is described. The transformer is immersed in an inner bath that nitrogen vapours are exhausted from, so the transformer windings can be cooled up to the temperature of the nitrogen triple point ($\approx$63K). The inner bath with transformer is surrounded by an external bath of liquid nitrogen at atmospheric pressure which absorbed practically all thermal flows from surroundings. Arrangement of this kind enables to measure heat losses of the transformer windings as low as 0,1W.
Relations among thermal flows, pressure and effective pumping speed was analysed and pumping system was designed according the results of the analyse. The pumping system permitted to stabilise temperature of the transformer at a value from the range 65–77K with a negligible temperature variation for a long time period.

THE SEGMENTATION – THE NEURAL NETWORK APPROXIMATION OF THE CCG CHARACTERISTICS OVER THE WIDE PARAMETERS RANGE

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The highly nonlinear Cold Cathode Gauge (CCG) characteristic can be satisfactory approximated by the neural networks. The approximation was performed in order to predict the CCG characteristics over the entire parameters working space. However there is a practical problem to perform a good enough approximation of the CCG parameters over the wide range of several decades, both in the CCG discharge current (typically from $10^{-10}$ A to $10^{-7}$ A), and vacuum chamber pressure (typically from $10^{-9}$ mbar to $10^{-6}$ mbar).

Neural networks transform the input space into the area $[0,1]$. This causes problems at the approximation process. The quality of approximation (approximation error) of small values is very poor compared to the large ones.

The commonly used practise is the logarithmation of the function. Neural networks are then used to approximate the secondary – logarithmed function. This practice proves to be good in classical approaches, but rather poor when used with neural networks. The neural network approximation introduces the approximation error, which is even greater when the anti logarithm is calculated to achieve the original values.

In the conjunction with neural networks two strategies have been studied. The first is the architectural approach where the different sections of the approximated curve is dealt by different parts of the neural network. At the second approach the approximated function is segmented prior to the approximation. The results of the later are presented in this paper.

TECHNOLOGIES IN VACUUM FURNACES FOR SMALL DISCHARGE TUBES MANUFACTURING

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Discharge tubes are hermetically closed cells filled with a suitable gas having the pressure between one and some hundreds milibar. Usually the cell housing is made of metal, glass or of ceramics and two electrically isolated electrodes are introduced into it. Greater types of gas discharge tubes (eg. lightning elements) are evacuated and than each one is separately filled by gas. Smaller ones (eg. different gas arresters) are manufactured in a special tool - many pieces together - in vacuum furnace, where at the end all samples are filled with gas simultaneously.
There are several interesting technics which need vacuum furnaces for making the final gas tube. These are as follows: degassing, feedthrough making, (re)melting and brazing in vacuum or in inert atmosphere, ceramic-to-metal joining. The mentioned techniques are important because the end electric quality of special cells directly depends upon performance of procedures, upon their ability to reach clean surfaces, vacuum tightness and demanded atmosphere in a closed tube.

The contribution represents the experiences on development of gas arrester cells; there are discussed different types of furnaces for manufacturing small discharge tube and are described the base finishing steps in mentioned furnaces.

**Key words:** discharge tubes, vacuum techniques, vacuum furnaces, gas arrester, glass-to-metal feedthroughs, gas encapsulation by brazing in inert atmosphere, ceramic-to-metal joints.

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**ULTRAHIGH VACUUM FLUXLESS METAL SOLDERING**

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At the closed-end channel electron multipliers, the metal soldering of the glass cathode sub-assemblies represents the last important phase of the vacuum transfer technique.

The fracture toughness, thermal cyclic resistance, extreme vacuum tightness of the soldered seals are the prerequisites of the correct functioning and operational lifetime of the channel photomultipliers.

The glass surfaces of the channel photomultiplier sub-assemblies are metallized with a vacuum evaporated Cu (or Ni or Ag-Cu-In) layer. In all cases, a thin Cr film which serves as the Cu or Ni, or Ag-Cu-In binding underlayer and a thin protective Au overlayer are evaporated.

The solder rings which are placed on the prepared metallization layers, consist of an indium eutectic alloy - InSn.

In order to achieve better solder wettability and thus better mechanical properties of the formed seal, two different metallization layer preparation processes were performed and investigated. The same materials were used to form the solder underlayer, but were treated under the different conditions. At first process the complete composition of glass sub-assembly with Cr, Cu (Ni, Ag-Cu-In), Au layer and InSn solder was vacuum baked for 9 hours at 360°C, while at the second experiments, the InSn solder was added to the metallized glass surface after the bakeout and melted at 130 °C for 1 hour.

For the samples that were prepared under the different conditions, the wettability and composition of the metallization layer, intermetallic compound formation, surface segregation of Cr, dissolution of Au, and the oxidation of the elements were investigated using SEM and HRAES.
Nano-sized yellow iron-oxide pigment Bayferrox 3920 has been exposed to oxygen plasma created in a plasma reactor with a volume of 30 dm³ with an inductively coupled RF generator with the frequency of 27.12 MHz and the power of 5000 W. The reactor was pumped with a mechanical rotary pump and the base pressure was about 1x10⁻² mbar. Experiments were performed at oxygen pressure of 50 Pa. The density of charged particles 1x10¹⁶ m⁻³ was determined with a double electrical probe and the density 1.6x10²¹ m⁻³ of neutral oxygen atoms in the ground state with a catalytic probe. The pigment was evenly distributed in a Petri dish with the diameter of 15 cm mounted in the plasma reactor. The samples were exposed to plasma for different periods from 30 to 600s. During the plasma treatment, a continuous change of the pigment colour from yellow through orange to dark-red was observed. Crystalline structure of the samples was tested with XRD. The valence state of iron in the samples before and after the oxygen-plasma treatment was examined with Fe K-edge XANES. Absorption spectra were measured in a standard transmission mode at the E4 station of HASYLAB synchrotron facility DESY (Hamburg, Germany) with 1 eV resolution. The XANES results show that the valence state +3 of iron does not change even in a prolonged exposure of the pigment to the highly oxidizing plasma. Rather, the change in pigment color can be ascribed to recrystallization of nanoparticles.

Optical emission spectroscopy analysis of inductively coupled RF oxygen plasma has been performed during plasma treatment of a Mylar foil. Plasma was generated within a glass tube with the internal diameter of 36mm with an RF generator with the frequency of 27.12 MHz and the output power of 300 W. The tube was pumped with a mechanical rotary pump. Experiments were performed at the oxygen pressure of 75 Pa. The density of charged particles was estimated in an empty discharge tube with a double electrical probe and was about 1x10¹⁶ m⁻³. The density of neutral oxygen atoms in the ground state was measured with a catalytic probe and was 3x10²¹ m⁻³. During the plasma processing of the Mylar foil the O atom peak at 777.4 and 844.6 nm, and the CO and H₂ peaks at 519.7 and 656.2 nm, respectively, have been measured simultaneously every 1s. The results showed that the O peak at 844.6 nm decreased at the first 2 second of
plasma treatment, reached a well-defined minimum, and increased with plasma treatment for next few second to reach a broad maximum at around 7s. The peak intensity was slowly decreasing with further increase of the treatment time. The behavior of the CO and H peaks was found reversed, i.e. the maximum of these peaks were detected when minimum in O peak was detected. The results were explained by oxidation of the Mylar foil during plasma treatment.

Keywords: plasma, oxygen, emission spectroscopy, polyester

ENERGY-RESOLVED MASS SPECTROSCOPY DURING THE DEPOSITION OF TiN, Ti(CN) AND TiC FILMS BY ION PLATING

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The mechanical properties, microstructure and average chemical composition of Ti carbonitride hard coating, deposited by means of the triode ion plating, strongly depends on many plasma parameters, but the most relevant are arc current, magnetic field and reactive gas partial pressures. All of them are related to the concentration of plasma constituents (ions, radicals,...).

To quantify these effects, we have thoroughly investigated the plasma during ion plating of Ti(CN) coatings by energy-resolved mass spectrometry. Studies were performed for mixtures of Ar + C2H2, Ar + N2, as well as different ternary mixtures of Ar + C2H2 + N2, in the pressure range from 0.12 Pa to 0.30 Pa, with a constant Ar pressure 0.12 Pa. We have found that there is a significant difference in mass spectra of Ar + N2 and Ar + C2H2 plasma. The latter ones are characterized by a high degree of dissociation. The main ionized species in Ar + C2H2 plasma during discharge is H+2,3, while the most abundant carbon-containing species is 12C+. Overall intensity of carbon containing ionized species (∑C,H+) drops with increasing arc current. This is in opposition to the intensities of other species (M, Ar) whose intensity follow the arc current. At high partial pressures of acetylene, in excess of 0.10 Pa, the ion flux is reduced and the evaporation of metal even stops. The Ar + N2 plasma does not show such strong dissociation, due to the much lower ionization cross section of nitrogen molecule. The most abundant nitrogen containing ionized species is N+ whose intensity is proportional to the plasma intensity, i.e. arc current. Magnetic field does not show simple relation with the plasma composition. But at fields lower than used for successful evaporation of titanium we observed a strong increase in overall ion intensity as well as of hydrocarbon (∑C,H+) ions.

On the basis of present understanding we believe that the metal ionization occurs within the arc, close to the crucible, while the ionization of reactive gases takes place within the whole volume of the plasma.
Dust grains in a space are charged by various processes. Impact of energetic ions leads to deposition of the positive charge in the grain and increases the grain potential and the electric field at its surface. The accumulated charge becomes spontaneously released when the electric field reaches a threshold. This discharging current is usually attributed to field ionization of a gas surrounding the grain or ion field emission and thus it would be a function of the surface potential only. However, our preliminary study using melamine formaldehyde spheres has shown that the discharging current depends strongly on the energy of primary ions. The present paper continues these investigations with motivation to understand the whole charging/discharging process. The experiment is based on the capture of single dust grain in an electrodynamic quadrupole. The trapped grain is exposed to the ion beam with different energies and its charge and surface potential are estimated from the frequency of its oscillations in the quadrupole. The charging/discharging currents are determined from temporal changes of the grain charge. Our results suggest that the grain charge is accumulated in a thick surface layer. The thickness of this layer depends on the mass and energy of primary ions. The process limiting the discharging current is probably diffusion of the holes created by primary ions toward the grain surface.

ROLES OF N AND O ATOMS IN A FLOWING N₂-O₂ MICROWAVE POST DISCHARGE IN THE SURFACE HEATING PROCESS OF SUBSTRATES

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In the recent years, there is more and more interest in plasma technology for bacteria sterilization because of its safe and low temperature action compared to the actual treatments. For sterilizing polymer parts of instruments used in medical environments, the material temperature must be lower than 60 °C.

The effect of a N₂-O₂ microwave flowing post discharge on the sterilization of different bacteria has been studied. Optimization of the process was performed varying gas mixture, source power, pressure and substrate temperature.

For a better understanding of the sterilization mechanisms in post-discharge environments, we have determined the absolute concentrations of N and O atoms by NO titration technique. The aim of this study is to determine the role of N and O atoms in the surface heating.

The density of N and O atoms has been measured by varying the microwave power from 40 to 100 Watt in a quartz tube of 5 mm int.dia., the gas pressure from 1 to 10 Torr at a constant gas flow rate of 1 L·min⁻¹ and for several O₂ percentages into N₂ from 0 (pure N₂ discharges) to 100 (in fact Ar-7%O₂).
Several holder materials were tested: pyrex, polymer, ceramic, Al, Ti, steel and brass whose temperatures were measured by using a thermocouple after a given post-discharge treatment. The role of each active specie: N or O–atom in pure N₂ or in Ar-7%O₂, has been specified. Also the effect of both N and O-atoms in N₂-O₂ post-discharges has been analyzed.

We have found that the holder heating was due to the O and N atoms recombination on the surfaces. It depends on the total N + O atom density produced in N₂-O₂ post-discharges and on the holder material. The brass support was heated about two times more than the other metals studied: after 30 minutes of treatment, 120 °C for the brass support compared to 60 –70 °C for the steel, Al and Ti surfaces. The pyrex, polymers and ceramics remained to the room temperature.
O ATOM DENSITY IN OXYGEN PLASMA BY Co PROBE
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Oxygen plasmas are used in several advanced technologies including the plasma etching, drilling, oxidation, cleaning, ashing and surface activation. Different technologies require application of plasma with different parameters. For etching and drilling, plasma with a high degree of ionization is needed, while for some other technologies, oxygen plasma with a low density of charged particles performs better. For sensitive treatments of polymer samples, a state of a gas with a negligible concentration of charged particles should be used. In such cases, it is much better to treat samples in post – discharges rather than in plasmas themselves. In systems used for sensitive plasma treatments, the most important parameter is the density of neutral oxygen atoms.

The density of O atoms in a plasma reactor was measured with 2 probes, a nickel and cobalt catalytic probes (FOCP probes), where the Ni FOCP was calibrated with NO titration. The measurements were performed at different pressure from 10 to 400 Pa. The results of neutral O density, gas dissociation and recombination coefficients of Co probe were explained by accommodation and recombination on the FOCP surface, taking into account collision phenomena in ionized gases.

EMISSION OPTICAL SPECTROSCOPY STUDY OF RF OXYGEN PLASMA VERSUS PRESSURE
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Optical emission spectroscopy of inductively coupled RF oxygen plasma has been performed in the range of oxygen pressure from 10 to 800 Pa. Plasma was generated within a glass tube of 36 mm internal diameter with an RF generator with the frequency of 27.12 MHz and the output power of 300 W. Plasma parameters were determined by means of a double electrical probe and a catalytic probe. The flow of oxygen was established in the tube using a mechanical rotary pump. Spectra were measured in the range from 200 nm to 1100 nm by means of a high-resolution optical fiber spectrometer. Observation was done through a quartz window mounted on the arm of the tube perpendicularly to the coil. At high pressures the main spectral features observed were atomic oxygen transitions at 777.4 nm and 844.6 nm, and molecular oxygen band at 762 nm. The atomic emission intensity shows a maximum at pressure of about 75 Pa, while molecular band intensity increases monotonic as the total pressure increase. By decreasing oxygen pressure other atomic (O) and molecular (O₂⁺) features appear in spectra, as well as H atomic lines and OH molecular band. The increase of atomic and molecular features follows different paths. The behavior of spectral intensity depending on total pressure is discussed in terms of different discharge modes. Lower total pressures promote ionization processes.
PHOTORESIST ASHING IN NITROGEN GAS USING FERRITE-CORE INDUCTIVELY COUPLED PLASMAS

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In order to fully take advantage of copper interconnects in semiconductor devices, low-k dielectric materials must be used to reduce interelectrode capacitance. However, standard ashing process using O2 plasmas in the conventional asher can damage the low-k layer through oxidation, resulting in the higher capacitance and thus defeating the purpose of using such a film. In the present work, we report the characteristics of ashing process using the N2 gas in the inductively coupled plasma system with a ferrite-core. By varying the process parameters such as gas flow rate and bias power, we have changed the ashing rate and low-k material (SiOCH) etch rate. With the bias power of 1000 W, the PR to low-k material etch selectivity of above 50 with minimal damage of low-k material was obtained. The unprecedentedly high value of PR-to-low k material selectivity may be caused by using the ferrite-core inductively coupled plasma, in which radicals of high density are supposed to be generated. A damage to low-k dielectrics during the ashing process was evaluated by using Fourier Transform Infrared Spectroscopy (FTIR) and by a relief etch with HF solution. This result may contribute to the potential applications of low-k materials in the copper interconnects technology.

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INVESTIGATION OF AN ATMOSPHERIC-PRESSURE RADIO-FREQUENCY CAPACITIVE PLASMA JET

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Usually plasma sources operated at atmospheric pressure are based on arc discharges and produce thermal plasmas with temperatures well above a few thousand °C. At reduced pressures large area discharges can be easily sustained in the glow discharge mode. However, large area glow discharges at atmospheric pressure are rather unstable. Recently, it was reported that an atmospheric-pressure plasma jet (APPJ) can be generated in a capacitive radio-frequency plasma source, resulting in a stable glow-like discharge with a rather low gas kinetic temperature.

We have constructed a large area atmospheric-pressure plasma jet (LAAPPJ) composed of two planar square electrodes. The gap width was 50 mm and the gap spacing can be varied between 0.5 and 2.5 mm. The LAAPPJ was operated with helium (purity 99.996%) at gas flow rates between 2 and 10 slm. The electrical properties of the discharge were studied by measuring the
voltage across the discharge and the discharge current simultaneously using a high voltage probe (Tektronix P5100), a current probe (Tektronix P6021 AC) and a digital oscilloscope (Tektronix TDS 3052B).

The investigations clearly showed that a stable glow-like discharge can be sustained within certain limits. There is a minimum voltage needed for the discharge, however, the required r.f power can be as low as 10 W. When the r.f power is raised above a critical level, the discharge suddenly turns into a filamentary arc. Lower limit as well as upper limit increases with gap spacing. The regime of stability does not depend on the gas flow rate.

**STUDY OF LASER ABLATION OF GRAPHITE-POLYMER AND POLYMER TARGETS USING CAVITY RING-DOWN SPECTROSCOPY**

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We performed laser ablation of different graphite-polymer (PPS) and polymer (Mylar type A), targets by means of a pulsed laser (XeCl, 308 nm, 15 ns). Laser fluence was about 1 J/cm². The target was rotated to avoid drilling. Induced plume is allowed to spread in vacuum at pressure of about 10⁻³ mbar. We have used cavity ring-down spectroscopy for absorption measurements in the plume within the spectral range (450-480) nm. The C₂ radicals were detected through the observation of Swan bands. Theoretical simulation of absorption spectra was used for determination of vibrational and rotational temperatures. Comparing to pure graphite laser ablation, much higher ro-vibrational temperatures were observed in the case of graphite-PPS target. In addition, observed C₂ density decreases as number of laser pulses increases, contrary to the case of laser ablation of pure graphite targets. In the case of pure polymer laser ablation, C₂ observation was possible only in the very early time windows (fast radicals). In later time periods, scattering on large (slow) particles gives dominant contribution to the cavity loss. Possibilities for monitoring of polymer laser ablation by means of the cavity ring-down spectroscopy versus other methods, such as optical emission spectroscopy will be discussed.

Keywords: laser ablation, graphite, polymers, absorption spectroscopy, cavity ring-down

**SECONDARY EMISSION FROM DUST GRAINS: A COMPARISON OF INSULATORS AND METALS**

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The secondary emission from metal planar surfaces is well understood. However, the space dust grains are composed of silicates, graphite or ice and their surfaces are highly curved. Moreover, they are often charged to non-negligible potentials and thus the secondary field emission yield
can be enhanced by the presence of a strong electric field above the grain surface. We have carried out systematic measurements of the secondary emission yield as a function of the energy of primary electrons for dust simulants of different diameters in a range from 1 to 10 microns. The study results show that the secondary emission charges the grains generally positively but the grain potential can reverse in a range of energies and grain diameters. The interpretation of these results, in terms of the penetration depth becoming comparable with the grain size, was confirmed by the Monte-Carlo model of charging process. However, the determination of model constants is rather difficult due to complicated molecular structure of used simulants. For this reason, we have repeated our measurements using metal spheres and compared the results with measurements on non-conducting grains.

**ELECTRON ENERGY DISTRIBUTION AND TRANSPORT IN HELIUM-XENON MIXTURES**

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The study of low-temperature weakly ionized plasmas in inert-gas mixtures has become increasingly important in various fields of technology, such as discharge lamps, gas lasers and plasma display panels. While the transport properties in pure inert-gas plasmas are rather well known, the interplay of the electron, ionic and neutral kinetics in multispecies gas mixtures is not yet fully understood.

In the present work we focus on plasmas in helium-xenon mixtures, and study the electron transport properties by the Boltzmann equation analysis, in the region of the Ramsauer-Townsend minimum (RTM) for elastic scattering of electrons on xenon neutrals atoms. The electron drift velocity $W$, i.e. mobility ($\mu=W/E$), and the diffusion coefficients namely, their ratio with mobility $D_T/\mu$ and $D_L/\mu$ are evaluated on the range of reduced electric field strengths $E/N$ that comprise the RTM and for different plasma compositions ranging from $k \in (0, 1)$, where $k = N_{He}/(N_{He} + N_{Xe})$. It was found that the presence of He, already in low concentrations ($k=0.05$), decreases significantly the mean electron energy and the $D_T/\mu$ coefficient. Conversely, at a given $E/N$ there is a value of $k$ for which $D_L/\mu$ is maximal. The most interesting region is in the immediate vicinity of the RTM, where the addition of He has the effect of increasing the anisotropy of the electron energy distribution function, raising the electron drift velocity over the values in both pure Xe ($k=0$) and pure He ($k=1$) plasmas. The appearance of the negative differential conductivity (NDC), i.e. the decreasing electron drift velocity for increasing $E/N$ in the RTM region is also examined and discussed.
Dust grains are present in many plasma as well as vacuum systems and/or in a space. Under specific conditions, their charging depends significantly on surface properties of a grain material. In cold plasma, charging is given mainly by electron attachment, nevertheless, when plasma becomes hot, other processes (secondary electron emission, field emission, etc.) take place. Emission properties of the grain surface could be modified by grain baking or by ion bombardment. We have chosen He$^+$ and Ar$^+$ ions for surface treatment and the observed influence on the surface properties is discussed in terms of secondary emission. A non-negligible shift of the secondary electron emission yield curve was measured after Ar$^+$ bombardment. Our study is carried out at the dust charging experiment dealing with a single dust grain electro-dynamically levitated in a 3D quadrupole trap. The grain can be exposed to the ion beam in the energy range of 200 eV – 5 keV and to the electron beam in the energy range of 200 eV – 10 keV. We compare metallic, pure melamine formaldehyde (MF) resin, and with thin metal films covered MF resin grains; all of them well spherically shaped.

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The neutral oxygen plasma is nowadays frequently used for etching of different coating surfaces. In this process the topmost layer of the binder is removed while the solid particles (pigments, fillers) remain intact. By analyzing the SEM micrographs of such surface, the state of dispersion of the pigmented coating may be quantified.

Organic polymers used as a binder in pigmented coatings have different chemical and physical properties and consequently, the cured polymeric film. Therefore, different etching efficiency may be expected on such samples.

The neutral oxygen plasma with the same plasma characteristics was used to etch various organic polymers. The etching efficiency was evaluated by the measurements of the reduction of the polymer layer thickness as a function of etching time. The chemical nature of the etched polymer layers was analyzed by the infrared spectroscopy.
The very low frequency (VLF) (<30 kHz) radio diagnostics is a well-known and powerful tool for the investigation of the ionized medium of the lower ionosphere. Disturbances that occur in the amplitude and phase stabilized VLF recordings emitted from known locations, indicate specific natural phenomena that change the characteristics of the propagation environment, namely, its electron density, electron temperature and ion composition. Disturbances of the ionized medium caused by solar flares, total eclipses, whistler-induced electron precipitation (WEP) and seismoionospheric effects appear on long time scale, while the ones caused by lightning-induced electron precipitation (LEP) occur on short time scale. Both kinds of disturbances have been recorded by the new AbsPAL (absolute phase and amplitude logging) receiver installed at the Institute of Physics, Belgrade (44° 38’ N, 20° 46’ E) in August 2003.

All the above mentioned effects have been detected on the VLF signals emitted at 24.0 kHz, on the west-east trace (NAA), along the 45° N parallel. Each effect yields information about the relaxation time of the disturbed propagation medium. Subsequently, relaxation times are connected with the effective recombination coefficients, characteristic for radiation and transport processes in the ionized medium. Comparing the known theoretically obtained rate coefficients for low temperature, weakly ionized plasma, with values estimated from VLF monitoring, the model of the lower ionosphere could be significantly improved. Presently, from the time-resolved recordings of the disturbances, the relaxation times for each of the effects observed have been estimated and, the corresponding effective recombination coefficients have been deduced. On the basis of these data, a model of the electron density vertical profile has been proposed.
LASER-ANNEALING OF SPUTTERED SILICON FOR WAFER-BONDING APPLICATIONS

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Sputtered silicon is being investigated as a bonding surface for transfer of pre-processed silicon layers to various insulating substrates including silicon oxide, quartz, and low temperature materials. Applications include opto-electronics, microelectronics, sensors, actuators and MEMS. Previous work has shown that the material appears quite suitable for low temperature processing. However, when annealing of bonded layers for thermal splitting or bond strengthening is required, gas trapped in the pores of the sputtered material is released at temperatures above 350 °C and destroys the bonded interface. The gas can be effectively driven out by thermal annealing at a temperature of 1000 °C. For device applications when processing temperatures before bonding must be kept below about 300 °C, this technique cannot be used. In the current work we have investigated laser-annealing of the sputtered silicon surface to remove trapped gases without significant heating of the underlying substrate. The effect of laser power density on the silicon surface is discussed and process details are reported. The experiments show that it is possible by overlapping laser-beam scanning to achieve optimum conditions for trapped gas removal and yet maintain a low roughness surface suitable for subsequent wafer bonding.

PLATEAU-TO-PLATEAU TRANSITIONS IN THE INTEGRAL QUANTUM HALL EFFECT IN InGaAs/InP

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InGaAs/InP and related InP based heterostructures are of great scientific and technological interest for their application in optoelectronic and electronic devices. There are also fundamental issues related to the existence and properties of two-dimensional electron gas (2DEG) at the heterojunction interface or in the quantum wells. The transport properties of 2DEG, especially those related to the quantum Hall effect continue to attract much interest both experimentally and theoretically. The steps connecting the quantum Hall plateaus become sharper with decreasing temperature, and according to the scaling theory, the reciprocal linewidth of the \( \rho_{xx} \) peaks and the maximum slope of \( \rho_{xy} \) between the quantized plateaus diverge as \( (\Delta B)^{-1} \sim T^{-K} \) and
(d\rho_{xx}/dB)_{max} \sim T^{-k}. The scaling exponent k contains information on the localization length and on the mechanisms of inelastic electron scattering.

We have studied the transitions between the low Landau index integral quantum Hall plateaus in 2DEG in liquid phase epitaxial In_{0.53}Ga_{0.47}As/InP modulation-doped heterostructures, with electron density and mobility of (0.4-4) \times 10^{11} \text{cm}^{-2} and (1-5) \times 10^{4} \text{cm}^2/\text{Vs} respectively. We have measured the longitudinal \rho_{xx} and Hall \rho_{xy} resistivities as a function of magnetic field in the temperature range 40 \text{mK} – 4.2 \text{K} in magnetic fields up to 20 \text{Tesla}. The scaling exponents were extracted from the temperature dependence of the linewidth of the \rho_{xx} peaks and from the maximum slope of \rho_{xy} between plateaus.

The scaling exponent for the low Landau index quantum Hall plateau-to-plateau transitions was found to have a value of K = 0.6-0.8. The relationship of the obtained experimental value to other data, some of them considered up to now universal in the literature, however some of them also strongly debated, is discussed in terms of the properties of the various material systems used in the experiments, and in terms of the possible differences in the relevant inelastic electron scattering mechanisms.

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MECHANICAL STRESS IN THIN FILM MICROSTRUCTURES ON SILICON SUBSTRATE

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In order to guarantee long term operation of microelectromechanical systems (MEMS), it is highly desirable to control mechanical properties of thin films. This may help to avoid catastrophic failures due to fracture, which is particularly critical in the assembly stage. Electrical device performances also degrade when high mechanical stress is presented. Furthermore, increase of etch and diffusion rates are as well observed in strained regions. High residual stresses in deposited thin films are undesired for mechanical structures because they cause buckling of thin silicon diaphragms and bending of cantilever beams without applying external force. On the other hand these deformations can be exploited to determine the state of stress in the thin film.

A study of residual stress in thin free-standing cantilevers and silicon membranes which are commonly covered by thin films such as thermal oxide, LPCVD or PECVD nitride was performed. High intrinsic stress was determined for deposited stoichiometric LPCVD silicon nitride, while for PECVD silicon nitride and thermal silicon oxide, lower values were determined. Deflection behaviour of membranes and cantilevers due to built-in stress in thin films were investigated in the scope of different bilayer structures which are commonly met in the fabrication of piezoresistive pressure sensor structures. By preparing samples with different final thickness of silicon membranes and deposited thin films, we observed how residual stress in thin films deforms the silicon membrane. Resulting undesired membrane deflection at zero
applied pressure increases the offset voltage and also introduces additional temperature dependency of the device response.

QUASICRYSTALS: POTENTIAL THERMOELECTRIC MATERIALS

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The problem of designing more performant thermoelectric devices has been and remains to be an important and vital problem of physics and technology. Its solution depends directly upon the process of obtaining more efficient thermoelectric materials. Such materials must possess high values of electrical conductivity and Seebeck coefficient as well as the lowest possible values of thermal conductivity. These requirements are in general of a contradictory character. Most quasicrystals exhibit very low thermal conductivity values (1-5 W/Km) with the signature plateau region around 50-150 K, which is typical of many amorphous materials. However, in the plateau region the thermal conductivity of quasicrystals can be almost an order of magnitude higher than the thermal conductivity of many amorphous solids. Even with the low thermal conductivity, quasicrystals have a relatively broad range of electrical conductivity [i.e., 0.01-5000 (Ωcm)^{-1}]. An advantage of quasicrystals is that the value of electrical conductivity as well as of the thermoelectric power can be modified by varying the composition without sacrificing the low thermal conductivity exhibited in these materials. For these reasons, we have done a systematic investigation of the electrical and thermal transport properties of different quasicrystalline materials, which up to now have not been the subject of investigations for thermoelectric applications. In this paper we focus on the interesting electrical and thermal transport properties of single-phase icosahedral Al-Pd-Mn.
ANNEALING EFFECTS ON GAN/ZNO/SI STRUCTURES PREPARED BY THE RF MAGNETRON SPUTTERING

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The growth and properties of GaN films have been intensively studied over the last few years due to their potential applications in optoelectronic devices as well as in high-power and high temperature electronic devices. However, the production of high quality GaN is complicated by the lack of a lattice-matched and industrially usable substrate material. In this study, we deposited the high-quality GaN thin film on ZnO/Si(001) substrates at room temperature by the RF magnetron sputtering method and investigated the effect of thermal annealing on the structural and optical properties. XRD and SEM coincidentally indicated that the thermal annealing up to the temperature of 900 °C did not significantly change the structural properties of the films. However, photoluminescence (PL) spectra showed that the UV emission became stronger and the strong and broad green emission appeared by thermal annealing at 900 °C. We have discussed the possible mechanisms.

THE ELECTRICAL ACTIVITY OF OXYGEN PRECIPITATES IN p-TYPE SILICON

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Czohralski-grown silicon is used in the semiconductor industry almost exclusively for manufacturing VLSI devices. Such a material contains small quantities (∼20 ppm) of dissolved oxygen, which can have a crucial effect on the properties of produced devices. In particular, electrical activity of oxygen precipitates formed upon thermal processing is extremely important. The present state of understanding is still incomplete and the experimental results differ from article to article.

Here, we present a study of deep levels in p-type Czohralski-grown silicon produced by oxygen precipitation carried out by deep level transient spectroscopy (DLTS). Activation energies and capture cross-sections of electrically active defects have been determined. Contrary to some literature data where oxygen precipitates are observed to produce very broad DLTS peaks, usually associated with extended defects, we have observed point-like DLTS peaks. The results are discussed in terms of the morphology of precipitates.
TEMPERATURE DEPENDED DEFECT PRODUCTION IN $\gamma$-IRRADIATED SILICON

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There have been much research efforts in recent years to improve the radiation tolerance of silicon detectors for the use in extreme radiation environments. The standard silicon used for such detectors contain very low oxygen and carbon concentrations. It has been found that increasing the oxygen concentration improves the radiation tolerance. Defect modeling which explains all the beneficial effects of oxygen is still incomplete. Recent works suggest that it is possible to convert $O_2$ to $O_3$ yielding to formation of electrically neutral defects such as $V_2O_2$ and suppressing the formation of deleterious radiation-induced defects such as VO.

In this work we report on deep level transient spectroscopy (DLTS) studies on n-type (100) silicon irradiated at different temperatures in order to increase oxygen dimer ($O_2^*$) concentration. The samples used for this experiment were irradiated at room temperature (RT), 150 °C and 350 °C using a $^{60}$Co gamma source ($\sim$1MeV) to a dose of 50 MRad. After RT irradiation deep level at 0.17 eV below the bottom of the conducting band assigned to VO center dominates the DLTS spectra, as expected. DLTS measurements after irradiation at 150 °C have showed that the formation of VO center is suppressed. After irradiation at 350 °C VO center has not been detected. Contrary to VO center, divacancy-related defects have been detected in DLTS spectra. Possible reactions that are believed to lead to oxygen dimer formations would be discussed in detail.

ENHANCEMENT OF MINIATURE AND SMALL-BANDWIDTH MICROMACHINED ANTENNAS USING HRS (HIGH RESISTIVELY Si) AND LCP (LIQUID CRYSTAL POLYMER) TECHNOLOGY

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Micromachined antennas have been fabricated on HRS (Resistivity: ~5000 ohm) and LCP (Liquid Crystal Polymer) substrates with the thickness of 380 µm and 50 µm. The antennas have the dimension of 29.15 mm², 23.32 mm², 12.04 mm², and 10.68 mm² each for the 5.8 GHz wireless LAN. As a result of fabricating the folded slot antennas with micromachined process, we have reduced the patch size of the half wavelength rectangular-shaped antenna by about 50%~70%. The process is as follows. First, the patterned HRS Si wafer and LCP are bonded at 270 °C, and then the copper layer placed on the top side of the LCP is etched. After that, via holes are fabricated and Al(3µm) is deposited on the backside of the LCP. At this time, the backside should be patterned by laminating DFR (dry film resist) in order to prevent Al etchant from flowing into deep via holes. In this paper, we have the bandwidth of 300 MHz for the micromachined folded slot antennas on COB (chip on board). A prototype scaled model has
been simulated and then composed by bonding TMM4 (dielectric constant $\varepsilon_r = 4.5$ and thickness $= 700 \mu m$). The prototype has the same value of the gain as the measured one at 5.775 GHz. The gain improvement of the folded slot antennas over half-wavelength antennas is 5 dB and the bandwidth of the folded slot antennas is about 100 MHz wider than that of the $\lambda/2$ antenna.

**ELECTRICAL AND OPTICAL CHARACTERIZATION OF PECVD THIN SILICON OXYNITRIDE FILMS**

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Materials with variable composition and wide variation in their electronic properties, such as silicon oxynitride films, offer the solution of a number of current existing problems in the development of nano-dimensional Si-based devices. Plasma-assisted deposition is the most suitable technique for film preparation due to good control of film thickness and easy doping of films by adding additional gases to the deposition process.

This paper reports the study on the electrical and optical properties of thin silicon oxynitride films deposited by rf plasma-assisted CVD process in a downstream reactor with Si(OC$_2$H$_5$)$_4$ as precursor and addition of nitrogen. All deposition were performed at substrate temperature of 200 °C, at a power of 80 W and at a DC-bias of 0, -120 and -600 V applied to the Si substrate. For film characterization, capacitance-voltage (C-V) and current-voltage (I-V) measurements, IR spectrophotometry and spectral ellipsometry have been applied.

IR spectra show the presence of small hydrogen in the films either as Si-H or Si-OH bonds. The IR vibrational bands, related to Si-O and Si-N bonds, reveal that the films are silicon oxynitrides. The ellipsometric data analysis confirms the oxynitride character of the films. Films deposited at DC-bias of -120 V possess the highest density of dielectric charge and interface traps. In this case the current density through the films is the highest and, correspondingly, the specific resistivity is the lowest. These results well correlate with the ellipsometric model, which reveals incorporation of unoxidized Si in the oxynitride network.
In this contribution we are presenting some results of the influence of the MESA etching treatment on electrical properties of GaAs detector testing structures.

The GaAs detector testing structures were made on semiinsulating GaAs (100) wafers with a guard ring: the main reason for the presence of a guard ring is that it limits the surface leakage current and corrects the electric field under the pixels. Our wafers were manufactured by different companies, all have the thickness of 500 µm.

Two types of Schottky contacts were tested: Ti/V/Au or Ti/Pt/Au deposited by e-beam evaporation. On the back side were ohmic contacts with Ni/Ge/Au multilayer metalization. Pixels with different area were tested, namely from 50 x 50 µm² up to 1000 x 1000 µm².

The electrical characterization of the pixels has been carried out in a probe station using a Keithley 237 High Voltage Source Measuring Unit controlled by PC through GPIB interface. The current obtained from I-V characteristics is given as current density for comparison of various pixel areas and materials.

The MESA etching was effected by a chemical solution consisted of the \( \text{H}_2\text{SO}_4: \text{H}_2\text{O}_2: \text{H}_2\text{O} \) system most commonly used as a surface polishing etchant. For shallow etching a chemical etchant with a very low etch rate was used

We have found out that shallow etching does not have influence on electrical properties of detectors. Deeper etching larger than 1.5 µm does increase the value of the reverse breakdown voltage by 100 to 200 V in dependence on pixel area. In addition the shallow etch can be used to reveal the leakage currents between two contacts.

Detailed results will be given.

VIBRATIONAL STUDY OF THE CRYSTALLINE PHASES IN (PEO)₉ZnCl₂ NANOCOMPOSITE ELECTROLYTE

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The ability of poly(ethylene oxide), PEO to form crystalline complexes with a variety of salts has provided a number of novel compounds in which to examine fundamental questions of structure and coordination. Addressing these fundamental questions has practical implications as well, because insight into cation-polymer and cation-anion interactions is critical to understanding the microscopic mechanism of ionic transport in polymer electrolytes. The
interaction of the cation with the polymer is especially important because the transport of ions in the amorphous phase of PEO-based electrolytes appears to be dynamically coupled to the segmental motion of the host polymer.

In this work we shall study electrolyte from polymer-salt compounds based on ZnCl₂. Polymer electrolyte (PEO)_xZnCl₂ has been prepared by sol-gel procedure. Goal was to implement it as electrolyte in rechargeable galvanic cell with nanostructured metal oxide electrodes. Nanocomposite polymer electrolyte was formed by adding 25 nm sized TiO₂ grains during the preparation procedure. The influence of added nanosize TiO₂ grains to polymer electrolyte was studied by comparative vibrational spectroscopic study of IR and Raman measurements.

**OPTICAL AND STRUCTURAL INVESTIGATION OF GaNₓP₁₋ₓ/GaP STRUCTURES FOR LIGHT EMITTING DIODES**

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GaNₓP₁₋ₓ alloy represents a novel semiconductor alloy that has attracted considerable interest as a candidate for realization of the light emitting devices (LED’s). Incorporation of small amount of nitrogen as a dopant into GaP can replace phosphor atoms and forms isoelectronic traps. These traps enable rare green radiative recombination in the indirect GaP. With even higher nitrogen concentration phosphor rich GaNₓP₁₋ₓ alloy has been obtained. With increased level of nitrogen concentration, nitrogen atoms form NNᵢ pairs, which shift light emission wavelength from green to yellow-red spectrum. The existence of quasi-direct band gap transition was predicted for GaNₓP₁₋ₓ alloy (x=3%) using large 512-atom pseudopotencial super cell calculation.

Structures with variable N content in GaNₓP₁₋ₓ layer (x = 0.006 – 0.023) were grown on GaP substrate (100) by low pressure MOVPE. For characterization of the structures several methods were employed for structural property (SEM- SE, CL and SIMS profiling) as well as optical property (absorption spectroscopy, electroluminescence, and Raman spectroscopy) investigation.

Based on this results GaNₓP₁₋ₓ/GaP structures containing different nitrogen concentration were grown on GaP substrate by MOVPE technique. Structural properties as well as electrical and optical parameters were measured and analyzed at different temperatures. This study is focused on the investigation of structural and optical properties of GaNₓP₁₋ₓ/GaP layers and GaNₓP₁₋ₓ/GaP MQW LED structures and devices.

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There has been great interest in the last years in gas sensors based on porous silicon. Recently, a gas sensing device based on hydrocarbons (CH\textsubscript{x})/porous silicon structure has been fabricated [1]. The porous samples were coated with hydrocarbons groups deposited by plasma of methane under argon atmosphere. We have experimentally demonstrated that the structure can be used for detecting a low concentration of ethylene, ethane and propane gases.

In this paper, the CH\textsubscript{x}PS\textsubscript{Si} structure has been used as a sensing material to detect CO\textsubscript{2} and H\textsubscript{2} gases. Sensitivity of the devices, response time and impedance response to different gas exposure (CO\textsubscript{2}, H\textsubscript{2}) have been investigated.

The results show that current-voltage and impedance-voltage characteristics are modified by the gas reactivity on the PS\textsubscript{CH\textsubscript{x}} surface.

In conclusion, the sensor shows a rapid and reversible response to low concentration of the gases studied at room temperature.


CHARACTERISTICS OF OHMIC CONTACTS TO n-TYPE GaN

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Gallium nitride based wide bandgap semiconductors find increasing applications in optical (LED’s, lasers, detectors) and microelectronic (high-temperature, high-power and high-frequency transistors) devices. However, the performance of GaN-based devices is limited by several materials and engineering problems, including the difficulty in making low-resistance, thermally stable metal contacts, especially to n-type GaN. To understand such contacts, and hence establish the major factors influencing electrical behaviour, studies of the microstructure of various contacts are required. These can then be linked with observed variations in contact resistance and morphology. Two models – low barrier Schottky or tunneling contact – have been proposed to explain ohmic behaviour of metal contacts to n-type III-nitrides.

Al, Au, Al/Ti and Au/Ti metal layers have been deposited by thermal evaporation onto Si-doped n-type GaN epitaxial layers (n≈2–4·10\textsuperscript{18} cm\textsuperscript{-3}) grown by metal organic chemical vapor deposition (MOCVD) on a c-plane sapphire substrate. We have investigated the interfacial reactions and electrical properties of the heated contacts by XTEM, EDS, FESEM, XRD and I-V characteristics, and the material reactions were related to the electrical properties for obtaining low resistance Ohmic contact. The samples have been annealed at 400, 700 and 900 °C for 10 minutes in vacuum.
Al single contact layers showed dendritic formations after high temperature annealing. The continuous Au film changed to an island-like morphology. X-ray diffraction examinations showed, that new TiN interface phase formed in Al/Ti/n-GaN contact at 700 °C. XTEM, EDS and XRD investigations of the same contact revealed that Au diffused into the n-GaN layer at 900 °C. The surface of Au/Ti contacts annealed at 900 °C have shown fractal-like structures.

Preliminary current-voltage characterizations showed that the as-deposited Al and Al/Ti contacts are Ohmic while the as-deposited Au and Au/Ti contacts are rectifying. Ohmic or rectifying behaviour of the annealed contacts depending on annealing temperature will be discussed for the above metallizations.

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OPTICAL PROPERTIES OF LIQUID PHASE EPITAXIAL In_{x}Ga_{1-x}As_{y}Sb_{1-y} ON GaSb

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The quaternary alloy In_{x}Ga_{1-x}As_{y}Sb_{1-y} lattice matched to GaSb substrate is a material of great interest for optoelectronic devices operating in the mid-infrared wavelength region (from 1.7 to 4.4 m). The potential applications in this wavelength domain are gas spectroscopy analysis, optical transmission through the atmosphere, and light-wave communication systems to mention but a few.

We have studied the optical properties of lattice matched (a/a < 1.5·10^{-3}) In_{x}Ga_{1-x}As_{y}Sb_{1-y} layers with 0 < x < 0.20 grown by liquid phase epitaxy (LPE) on (100) oriented GaSb. The grown In_{x}Ga_{1-x}As_{y}Sb_{1-y} layers were characterized by X-ray diffraction (XRD) and the composition of the epitaxial quaternary layers was determined by an electron microprobe analyser (EMPA) in the wavelength dispersive mode (WDA).

The variation of the near band gap optical absorption coefficient with photon energy was determined from the analysis of room temperature optical transmission measurements. Band gap energies down to 0.55 eV were obtained at the highest In composition achieved (x = 0.204). The dependence of the band gap on the composition exhibits smaller bowing than predicted by recent theoretical calculations based on the correlated function expansion technique. The K coefficient determined from $\alpha = K(hv - E_g)^1/2/hv$ decreased with increasing x. Near and below the fundamental absorption edge an exponential absorption tail corresponding to the Urbach rule was observed which is ascribed to the effects of the random potential due to the alloy disorder.

Low temperature photoluminescence measurements were also performed on some of the layers, and preliminary results will also be presented.
Amorphous SiGe layers were sputtered from a SiGe polycrystalline target on both n- and p-type Si substrates (frequency – 13.6 MHz, bias – 1.5 kV) in Ar + plasma at 2.0·10⁻³ mbar pressure at room temperature. The Ge content of the target was about 6 %, but according to Rutherford backscattering measurements performed on similarly prepared samples, the Ge content in the sputtered films was only 4 %. Two different methods of Si surface preparation was used before sputtering. The "Acetone" treatment consisted of consecutive boiling the wafer in new clean acetone three times for 5-5 min. The "HF" treatment consisted of two steps: 1) Cleaning in 1:1 solution of H₂SO₄ and H₂O₂ for 30 min. 2) Etch in 1:20 solution of HF and H₂O for 1 min.

To study the effect of the layer thickness, SiGe layers with thickness in the range of 240-900 nm were deposited onto p-type substrates using the "Acetone" treatment. The effect of hydrogenation was also studied introducing 0.4 % H⁺ into the plasma during deposition for "Acetone" treatment and film thickness of 900 nm (both n- and p-type substrates). The effect of "Acetone" vs. "HF" treatments was studied also on both n- and p-type substrates with a layer thickness of 900 nm.

Although a significant influence of the preparation was observed on the electrical behaviour, the structures without hydrogenation exhibited similar specific features. As a common picture, the current-voltage (I-V) characteristics consisted of four different parts. The first part at low biases showed ohmic behaviour and was connected with a parallel conductance due to hopping mechanism. The central, most abrupt part is attributed to a Schottky barrier formed by Al on the amorphous layers. The third part exhibited a near-linear logI-V relationship with a smaller slope at relatively high biases, i.e. the current was limited by space charge in the amorphous layer. In the fourth part of the characteristics at high biases, the current was limited by the series resistance.

With increasing thickness of the amorphous SiGe layer, the I-V characteristics of the p-type structures ("Acetone" treatment) became worse: the current due to hopping conductivity increased and the part of I-V characteristics limited by space charge became more dominant. The "HF" treatment increased the apparent Schottky barrier height for p-type and decreased for n-type structures.

The hydrogenated amorphous structures exhibited I-V characteristics with very weak temperature dependence. At low currents the I-V relationship was exponential, which saturated at higher current levels, where the I-V characteristics exhibited a quadratic dependence of the current on the bias indicating space-charge limited current.
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DIFFUSION PROFILE OF LITHIUM INTO HIGHLY RESISTIVE p-TYPE SILICON

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In this work, the effects of temperature and time of diffusion on the lithium profile into p-type high resistive silicon have been investigated. The high purity lithium (Li) metal (99.995 %) was deposited onto p-type o silicon surface and thermally diffused into the bulk at a vacuum about $2 \cdot 10^{-6}$ Torr. The four probes technique has been used to determine the diffusion profile of Li impurities into silicon. The scanning electron microscopy (SEM) was used to measure the diffused junction depth ($X_j$). The lithium diffusion constant $D_{Li}$ was then extracted using the measured surface concentration $N_{Li}$. Thus, the variation of $D_{Li}$ as a function of diffusion temperature has been determined. Simulated profiles have been obtained by means PC1D calculate tool. A good agreement has been found when the results, simulated and experimental profiles, were compared with the literature values.

MOBILITY TRANSIENTS IN VACUUM TOPTAXIAL Ag$_2$Se THIN LAYERS

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Ag$_2$Se thin epitaxial layers were grown under vacuum conditions. The starting components were evaporated subsequently on various substrates. Than an annealing period in vacuum chamber followed in order to obtain topotaxial layers, which were either poly- or monocrystalline ones, depending on the substrate material.

The small gap Ag$_2$Se binary alloy shows semiconducting properties at low temperatures. However, similarly to some other binary chalcogenides, it undergoes a first order phase transition at about 133 °C. Metallic-like behavior is supposed at higher temperatures. We have shown that the temperatures of the phase transitions are different in heating and cooling regimes, though the transformation is reversible. These transitions are reflected also by Hall measurements not only in the resistivity, but also in electron concentration and electron mobility values. These parameters have been measured and are discussed in this paper.

The mobility shows a sudden decrease at the critical temperature in heating conditions and a sudden increase in cooling conditions. The phase transformation shows a hysteresis, thus four
temperature points were especially chosen for further comparisons of the measured parameters. Mobilities just before the "drop", just after the "drop" in heating conditions, and mobilities just before the "jump" and just after the "jump" in cooling conditions. The great number of samples of both types (mono- and polycrystalline) allowed us to obtain "concentration" dependence of the above "mobilities".

THE EFFECT OF VACUUM HEAT TREATMENT ON THE DIAMOND SURFACE CONDUCTION

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Monocrystalline epitaxial diamond layers were grown on Ib diamond (100) surfaces. Low level of boron doping was performed in microwave plasma reactor during the growth process. Two and four point probe electrical and low excitation level cathodoluminescence measurements were carried out first on as grown samples, both on epitaxial layers and monocrystalline bulk substrates. The samples were then treated in vacuum, in evacuated quartz tube in subsequent heating periods at 700 °C and then at ~900 °C. The same measurements were then performed in order to estimate the changes in the surface conductance due to the vacuum annealing. It was demonstrated that the surface resistance of the epitaxial layers increased several orders of magnitude, up to the semi-insulating level. This effect can be explained on the basis of earlier observations concerning the effect of hydrogen incorporation on the diamond surface and in the near surface region. The hydrogen could be incorporated during the growth process or on the air after the samples were taken out from the reactor. Similar resistance increase can be obtained when the surface is oxygen treated. The same effect was not observed on the HTHP bulk crystals. The cathodoluminescence measurements did not show significant differences between the as grown and vacuum annealed surfaces. This can be due to the complicated defect structure and the genuine inhomogeneity of the crystals. Furthermore the depth of the electron penetration of the exciting electrons can be higher than the thickness of the hydrogen disturbed surface layer.
THE EFFECT OF VACUUM HEAT TREATMENT ON THE DIAMOND SURFACE CONDUCTION

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