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**5th EEIGM
International Conference
on Advanced Materials Research
EEIGM / AMASE / FORGEMAT**



**November 4-5, 2009
EEIGM Nancy - France**

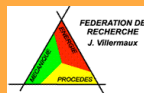


BOOK of ABSTRACTS



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Programme

Day 1: 4th November 2009

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20:00 Gala Dinner (Nancy City Hall)

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15:00-15:30 : Closure Meeting		

Explanation of colour scheme

	Characterization and Process
	Ceramics
	Metallic materials
	Composite materials
	Organic materials

Chairmen

	F. Mücklich and J.Ph. Bauer
	A. Mateo and H. Vehoff
	M. Anglada and Ph. Lagrange
	R. Joffe and S. Diebels
	W. Possart and B Jamart

Scientific Committee

M. Anglada
B. Jamart
F. Mücklich
J. Varna

Organizing Committee

Z. Ayadi
T. Czerwiec
C. Denis
C. Duclou
D. Gigoux
D. Horwat
D. Petetin

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Oral communications

Characterization and process

CONTROL OF RESIDUAL STRESSES AND DEFORMATIONS IN THE AUTOFRETTAGE PROCESS CHAIN OF HIGH PRESSURE COMPONENTS

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Abstract :

Increasing system pressures within hydraulic systems, e.g. in modern diesel injection systems, lead to challenging manufacturing procedures. Processes to increase the fatigue strength of high pressure components are nowadays commonly found in the manufacturing process chains. The hydraulic Autofrettage is one of those state-of-the-art processes to selectively induce compressive residual stresses in the failure-critical areas of the components (e.g. bore intersections). Although the effect of the Autofrettage for the fatigue strength of the machined component is subject to research activities, there have so far been very little activities to describe and examine the behavior and effect of residual stresses and deformations along the process chain of the processed component. As the Autofrettage process and its influence on the design of modern process chains for high pressure components will be one of the research topics of the Chair of Manufacturing technology, this paper gives an overview of the first activities in this area. The machined component undergoes a number of different manufacturing steps like e.g. heat treatment, machining, de burring or forming which all lead to unknown initial residual stress distributions as well as deformations on a macro and micro scale. The residual stress distribution along every step of the process chain will be described with Finite Element methods and iteratively developed with corresponding measurements. The objective is to describe and verify superposition effects of residual stresses and possible deformations caused by relocation effects due to machining processes after the Autofrettage.

STUDY OF A NEW LOW ELASTIC NI-FREE TI ALLOY BY INSTRUMENTED NANOINDENTATION

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Abstract :

Nowadays, β type Ti-based alloys have been developed for load transfer clinical applications due to their superelasticity, shape memory effect, low elastic modulus and high damping capacity. The objective of the present work is to achieve a material with shape memory properties and/or low elastic modulus. For that, the influence of cold work on the thermoelastic martensitic transformation and elastic modulus of the Ti-16.2Hf-24.8Nb-1Zr alloy has been investigated to determine the key factor that promotes the desired properties. A vacuum arc melted button of the alloy was heat treated at 1100 °C during 1.5 hours and quenched with a mixture of ethanol/water at 0 °C. Samples of the alloy were cold rolled from 5% up to 99% and finally, microstructurally and mechanically characterized.

The elastic modulus for each condition as well as the reversibility percentages were evaluated by instrumented nanoindentation using a spherical tip. A higher proportion of thermoelastic martensite was found in the low cold work percentages compared to the as received alloy as it was observed by optical and TEM microscopy. A decrease in elastic modulus was observed when increasing the cold work percentage. The lowest value was found in the 99% cold work condition with 44 GPa, value closer to that of cortical bone.

Cyclic nanoindentation tests show an increase in the reversibility percentages in the cold worked condition compared to the as received alloy.

INVESTIGATION OF HDPE UNDER TENSILE TEST BY BACKSCATTERED POLARIZED LIGHT

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Abstract :

Two in-situ non-invasive optical techniques can be used in order to characterize the deformation of High Density Polyethylene (HDPE) under uniaxial tension. Indeed, a videoextensometer (Videotraction®) gives access to the behavior law of the material (true stress/true strain relationship) at constant applied true strain rates. In a second step, the incoherent steady-light transport (ISLT) characterizes the evolution of the microstructure thanks to the development of objects that scatter light over a few cubic millimeters. Its application is limited to turbid materials and therefore is especially well adapted to semi crystalline polymers (PSC). The coupling of these techniques in real time gives highly complementary information and the main deformation stages can be then discriminated dynamically [1, 2]. The experimental measurement of the backscattered polarized light is acquired during a test and treated in three stages. First, the decrease in spatial intensity of the scattered light is modeled by radiative models in the diffusion approximation [3] which depends on the mean free path of photons inside the medium. Secondly, the angular variation of the backscattered intensity at different radial positions from the laser spot (anisotropy) is analyzed and it is shown that the appearance of an increasing anisotropy with strain means that scattering objects are progressively deformed along the tensile axis. This confirms all experimental studies that indicate a strong fibrillar microstructure and a fragmentation of crystalline lamellae [4]. At last, the amplitude of polarization effects is investigated, through the construction of Mueller matrices. These results are directly correlated to the size of the scattering objects. As PSC usually display a multi-scale structure with the coexistence of intimately related crystalline and amorphous phases, the connection between these phases and the objects that are responsible of the light scattering is not easy. Some considerations on this topic will be given in the communication.

THE EFFECTIVE METHOD BASED ON IR ANNEALING FOR MANUFACTURING NOVEL CARBONE NANOCRYSTALLINE MATERIAL AND MULTIFUNCTIONAL METAL POLYMER NANOCOMPOSITES

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Abstract :

The discovery of novel allotropic carbon form fullerene attracts attention to thermal polymer treatment under IR-irradiation that is considered as a perspective method for manufacturing carbon nanocrystalline material (CNM) and metal-polymer nanocomposites (MPNC). Under IR-irradiation, the thermal treatment of composites based on polymers (polyvinyl alcohol, polystyrene, cellulose, polyethylene terephthalate, polyacrylonitrile) and metals Fe, Cu and their compounds is an inexpensive method of obtaining the nanocomposite. For obtaining CNM and MPNCs, IR-annealing method is first used. Besides, polymers are treated by IR laser, to synthesize MPNCs and CNM. Cu compounds are introduced into the polymer solution. It is determined by mass-spectrometry that H and H₂ evolved under IR radiation reduce Cu²⁺ to Cu⁰. In the polymer matrix, Cu nanoparticle aggregation is complicated by the complex polymer activity, and Cu nanoparticles in the polymer are uniformly distributed. It is defined by X-ray that, in Cu/polymer nanocomposite, Cu (dCu₁₁₁=2,100 Å; dCu₂₀₀=1,817 Å) nanoparticles 10 nm in size are formed at 1400C.

It should be noted that the repeated annealing of the composite at 1400C in the air does not lead to forming copper oxides. Cu nanoparticles are stable in the air on heating since the strong Cu/polymer complex is formed. For manufacturing a fuel cell, nanocomposite Cu/C as the catalysts for oxidizing methanol at room temperature and obtaining hydrogen can be used. It is determined that after methanol exposure at 25°C in presence of nanocomposite Cu/C in a closed flask, formaldehyde (CCH₂O=49 mas.%) solution in methanol is formed. Thus, the nanotechnology enable one to manufacture the economically effective novel CNM and MPNC with perspective properties.

EVOLUTION OF STRUCTURAL AND PHYSICAL PROPERTIES UPON ANNEALING OF SPUTTER-DEPOSITED $Zr_{0.84}Y_{0.16}-O_2$ FILMS INCORPORATING COPPER AND PALLADIUM NANOPARTICLES

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Abstract :

The chemistry of ternary and quaternary metal oxides is of great interest as it can be exploited to tune the properties of the synthesized material with great freedom. Physical vapor deposition (PVD) processes widen the range of chemical, structural and morphological states that can be achieved (see for instance [1, 2]). PVD can produce materials with unusual properties because the high cooling rates used in the low temperature condensation of a thin film generates out-of-equilibrium states. Different paths towards the equilibrium state can be achieved upon annealing such metastable structures in air, depending on the reactivity of the film constituents with oxygen. The present paper reports on the incorporation of palladium or copper into $Zr_{0.84}Y_{0.16}-O_2$ thin films and on the change in the physical and chemical properties of such films after annealing at 300°C in air. Besides potential applications of functionalized zirconia-based materials in electrochemistry and as biomaterials, the present study sought to provide insights into the chemical states of the incorporated metals.

Me-incorporated $Zr_{0.84}Y_{0.16}$ oxide thin films (Me: Cu or Pd) were synthesized by reactive magnetron co-sputtering. The film structural evolution due to metal content increase was shown: Me-doped nanocrystalline YSZ; Me-doped amorphous oxide; metal nanoparticles embedded in the amorphous oxide matrix. The influence of thermal treatment in air (2 h at 300°C) on the film structure was investigated. Thermal treatment was found to promote copper oxide formation and the segregation of very fine Pd particles. XANES analysis at the Cu-K edge showed that Cu is bonded to oxygen and Zr(Y) in Cu-doped amorphous YSZ; this state was not affected by the thermal treatment. The XANES and resistivity analyses disclosed that the Cu nanoparticles likely have oxidized surfaces.

ELASTICITY MAPPING OF SINGLE PHASES IN POLYCRYSTALLINE METALS USING ATOMIC FORCE ACOUSTIC MICROSCOPY

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Abstract :

The elastic properties of polycrystalline materials such as structural metals or ceramics depend on the elastic constants of the different phases and on the grain orientations. Often the constituents are not available as macroscopic samples. Therefore techniques are needed to probe elastic properties on a micro- or nanoscale. Atomic force acoustic microscopy (AFAM) combines atomic force microscopy with ultrasound. It is a contact resonance spectroscopy technique allowing one to measure elastic properties of surfaces with a local resolution in the nm range. The elastic constant measured with such techniques is the so-called indentation modulus which accounts for the normal and shear deformation in the tip-sample contact zone. We imaged the contact resonance frequency and contact stiffness distribution in Ti-6Al-4V which is a very common titanium alloy. The alloy consists primarily of two different crystal structures: the hexagonal close-packed (hcp) α -phase and the body-centred cubic (bcc) β -phase. These phases were clearly visualized by AFAM. The bulk mechanical properties of titanium alloys depend strongly on the phase configuration that has been formed during heat treatment. Quantitative evaluation of AFAM measurements indicated an increasing indentation modulus of the β -phase with decreasing amount of vanadium. Ultrasonic bulk measurements, however, showed a decreasing macroscopic modulus with increasing temperature. The results clearly indicate that the predominant factor attributing to the change in modulus of Ti-6Al-4V is the volume fraction and not the chemical composition of the β -phase. For evaluation of AFAM data we used analytical models and finite element analysis of the vibrating cantilever.

FIB-TOMOGRAPHY AND 3D IMAGE ANALYSIS FOR QUANTITATIVE CHARACTERIZATION OF MICROSTRUCTURES

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Abstract :

The properties of many modern materials are strongly influenced not only by their chemistry, but also by their microstructure. As these microstructures often consist of complex constituents, determination of the real 3D shape becomes increasingly important.

In the recent years, FIB-tomography has been developed for 3D imaging of materials microstructures. It is a serial section technique, where the region of interest is consecutively sliced with a focused ion beam (FIB) and the cross section is imaged with a scanning electron microscope (SEM). Both techniques are combined in a FIB/SEM Dual Beam Workstation. All contrasts known from conventional scanning electron microscopy may be used. Usually images are taken in backscattered electron or secondary electron contrast (best resolution), but with new generation instruments even EDS (energy dispersive x-ray spectroscopy) and EBSD (electron backscatter diffraction) mappings are possible within a tomography for chemical and orientation or phase contrast. With FIB-tomography, a resolution of a few nanometers is achievable but on the other hand, larger volumes up to $(100\mu\text{m})^3$ can be studied as well and so this method is suited for a great range of microstructures. For 3D analysis, the suited tomographic method is of great importance and FIB tomography proves to provide high resolution images of relatively big volumes. With help of modern software, microstructures can be described quantitatively and effective properties can be simulated out of real tomographic data. This leads to a better understanding of formation of microstructures and allows the connection to the materials properties.

Ceramics

PREPARATION AND FIRING OF A TiC/Si POWDER MIXTURE

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Abstract :

This paper describes how the preparation and heat treatment of TiC/Si powders influences the phase reactions during firing. The powders are prepared by milling and some effects of powder preparation are discussed. A solid state displacement reaction according to: $3\text{TiC} + 2\text{Si} \rightarrow \text{Ti}_3\text{SiC}_2 + \text{SiC}$ is a priori expected to take place during heat treatment. The firing procedure is investigated with respect to the effect of heat treatment time and temperature on the phases produced, especially Ti_3SiC_2 . Samples were heat treated in a graphite lined furnace. Heat treated samples are analysed by x-ray diffraction, scanning electron microscope and energy dispersive spectroscopy. Ti_3SiC_2 , TiC and SiC are dominant in the final products. The highest amount of Ti_3SiC_2 is achieved for short holding times (2-4 hours) at high temperatures (1350-1400°C). Ti_3SiC_2 appears to decompose at elevated temperatures or extended times, through a $\text{Ti}_3\text{SiC}_2 \rightarrow \text{TiC} + \text{Si(g)}$ type reaction. The activation energy of Ti_3SiC_2 phase formation is determined to be 289 kJ/mol, using the Mehl-Avrami-Johnson model.

LOW TEMPERATURE DEGRADATION OF ZIRCONIA

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Abstract :

Yttria doped zirconia is widely used in engineering in different applications of large technological interest such as solid oxide fuel cells, thermal barrier coatings and body implants. In all these applications, service conditions are very different, but always one of the main concerns is durability because of degradation with time of mechanical and functional properties. In particular, zirconia doped with 3 mol % Yttria (3Y-TZP), which is used in biomedical applications, has a high mechanical resistance and good fracture toughness, being both properties a result of a very small average grain size and of shielding of the crack tip by the t-m transformation that is activated in the crack process zone. However, 3Y-TZP is prone to transform spontaneously to monoclinic at the surface in humid environments at intermediate temperatures around 250 °C, as well as, very slowly, at room temperature. Here we have analysed the response of tetragonal and cubic grains to nanoindentation and the microstructural and mechanical changes that take place during exposure of 3Y-TZP in contact with water vapour at 131 °C for different periods of time. The analysis is carried out by using nanoindentation, atomic force microscopy (AFM), micro-Raman, FIB-tomography, SEM and TEM. A model of thin film is applied to the degraded surface layer in order to extract the elastic modulus and hardness from nanoindentation measurements at different depths. These values are discussed in terms of the microcracks observed at the near surface by FIB-tomography. Finally the effect of grain size on hydrothermal degradation, mechanical behaviour under contact loading, mechanisms of hydrothermal degradation and methods for preventing it based on surface modification are briefly described.

DEVELOPMENT OF HIGH-FLUX ZEOLITE MEMBRANES FOR CO₂ REMOVAL OF SYNTHESIS GAS

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Abstract :

Zeolite membranes are potential candidates for carbon dioxide removal of synthesis gas, which is composed mainly of hydrogen gas and carbon monoxide. Our group is world-leading in the fabrication of high-flux composite membranes based on a thin film of MFI zeolite on the top of a porous alumina support with graded porosity. To enhance the poor selectivity of MFI membranes for the separation of CO₂ from H₂, we have investigated two modification procedures, namely: the introduction of barium ions in the zeolite structure by ion exchange and grafting of amine groups inside the zeolitic pores. Both methods reached a maximum separation factor of 6 with corresponding CO₂ permeances around 10×10^{-7} mol/m²·s·Pa in presence of water but at two different temperatures, 22 and 60°C, respectively. The achieved separation performances are considered to be sufficient for the targeted application. However, the method of film deposition originally developed on disks has to be transferred to a support geometry with higher membrane area per unit volume before the technique can be applied in industrial separation processes. Crack formation in the zeolite film during thermal cycling due to the different thermal expansion properties of the zeolite and the alumina porous support is another issue that needs to be solved: a concept of all-zeolite membranes is proposed.

INTERCALATION OF LITHIUM-EUROPYIUM ALLOYS INTO GRAPHITE

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Abstract :

According to their chemical compositions, the molten lithium-europium alloys react with graphite, leading either to EuC_6 binary compound or to a novel ternary graphite intercalation compound, whose chemical formula determined using nuclear microprobe is $\text{Li}_{0.3}\text{Eu}_{3.3}$. For this first stage phase, the intercalated sheets are poly-layered with a Li- Eu-Eu-Eu-Li c-axis stacking sequence. Using Mössbauer spectrometry, it has been established that europium presents a valence of +II in the ternary, so that its electronic configuration is $[\text{Xe}] 4f^7 6s^0$, corresponding to the $8S_{7/2}$ state. Consequently, europium possesses a strong magnetic moment, that confers very remarkable magnetic properties on the ternary. Its magnetisation and magnetic susceptibility versus temperature have been measured and the qualitative interpretation of their variations is proposed, using a previous model established in the case of EuC_6 binary, for which the intercalated sheets are strictly mono-layered. Ferromagnetic interactions between europium cations belonging to near planes are observed, while antiferromagnetic ones appear between europium ions belonging to the same plane.

Metallic materials

EMBRITTLEMENT MECHANISMS AT HIGH TEMPERATURES FOR A C-MN STEEL WITH HIGH RESIDUALS CONTENT

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Abstract :

Transverse cracking in the surface of semi-finished products is a problem related to the continuous casting steelmaking route. It is believed that cracks form during the unbending operation which is carried out at temperatures for which most steels exhibit poor ductility.

This behaviour is related to the appearance of different embrittlement mechanisms which mainly depend on the thermomechanical conditions and chemical composition of the steel. In order to characterize the embrittlement mechanisms associated with the presence of residual elements, a C-Mn steel produced from scrap recycling was evaluated in the as-cast and as-rolled conditions. Hot tensile tests were performed to assess the hot ductility and characterize the embrittlement mechanisms of the aforementioned steel. In addition to the initial condition of the material, i.e. as-cast vs. as-rolled, the effect of the thermal cycle prior to deformation was also evaluated. The %RA (reduction area) vs. temperature curves showed ductility troughs starting in the austenite single phase region and extending until the lowest testing temperature. However, the ductility drop took place at higher temperatures for the as-cast condition than for the as-rolled one. The fractographic analysis of the samples showed different embrittlement mechanisms depending on the thermal cycle. In-situ melted samples showed interdendritic fracture associated with the microsegregation that took place during solidification. When samples were reheated at low temperatures, i.e. 1100°C, interdendritic features were still apparent because the reheating temperature was not high enough to homogenize the composition. On the other hand, high reheating temperatures, i.e. 1330°C, gave rise to intergranular embrittlement probably due to the intergranular segregation of the residual elements and/or impurities

HIGH CYCLE FATIGUE OF METASTABLE AUSTENITIC STAINLESS STEELS

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Abstract :

Metastable austenitic stainless steels are currently used in applications where severe forming operations are required, such as automotive bodies, due to its excellent ductility.

They are also gaining interest for its combination of high strength and formability after forming. The biggest disadvantage is the difficulty to predict the mechanical response, which depends heavily on the amount of martensite formed.

The martensitic transformation in metastable stainless steels can be induced by plastic deformation at room temperature. In this research, the martensitic transformation was provoked by means of torsion testing. Several torsion angles were selected to achieve different percentages of martensite at the surface of the specimens. The next step was to evaluate their effect on the fatigue life of the steel. Fatigue testing in the high cycle regime was done at different levels of mean stress.

As a conclusion, the presence of martensite in the surface of the specimen led to an increase of the fatigue life when high mean stress was imposed. By contrast, at lower values of mean stress, martensitic transformation has no positive effect on the fatigue life.

CONTINUOUS COOLING TRANSFORMATION DIAGRAMS FOR 6XXX ALUMINIUM ALLOYS

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Abstract :

Alloys of the 6XXX series based on the Al-Mg-Si system took first place in production among all aluminum alloys. The only operation of heat treatment of these alloys prior to extrusion is homogenizing annealing. The decomposition of solid solution with precipitation of Mg₂Si phase particles occurs during cooling from homogenizing temperature. The aim of cooling rate regulation is generation of optimal density of Mg₂Si precipitations. For consecutive extrusion it needs, on the one hand, the maximal depletion of solid solution to decrease extrusion pressure and, on the other hand, the possibility of dissolution of Mg₂S precipitations during short-time hot extrusion to achieve the required mechanical properties of extruded products after ageing. Diagrams of transformations are used for studying of solid solution decomposition. It should be noted there is much more information in literature about time-temperature transformation diagrams. In our case, when we need to get information about kinetics of solid solution decomposition in continuous cooling conditions, the time-temperature transformation diagrams may give corrupted information. In this work the continuous cooling transformation diagrams in wide range of cooling rates for some 6XXX series alloys were built by changing of hardness of aged specimens after different cooling rates from homogenizing temperature. The temperatures of beginning and ending of solid solution decomposition was determined as well as effect of alloying elements – manganese and copper.

MECHANICAL PHENOMENA IN BULK METALLIC GLASSES

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Abstract :

Modern Bulk Metallic Glass (BMG) forming alloys represent a new class of metallic materials with unique properties in comparison to their well-established crystalline counterparts. The most extraordinary features are a net-shape forming ability, similar to polymer processing, and a high strength to high elastic strain limit. Research activities focused on processing and characterization show different phenomena assessed by studies with several representative alloys. Developed processing techniques that incorporate special restrictions like processing atmosphere and heat balance to ensure the frustration of the transformation to the thermodynamically favoured crystalline state [1] are presented. Thereby predominantly advantageous is the absence of this first order phase transformation that results in a very little shrinkage during solidification. Mechanical characterizations of BMGs were conducted in terms of size and strain-rate effects. Although metallic glasses are known to show very limited plasticity, experiments proofed the dependence of plastic deformation to the ratio of specimen size versus fracture process zone size. Shear-band formation, as typical fracture behaviour, as well as a transition to a hardening regime was observed. Compression tests within a large range of strain-rates, i.e. from quasistatic conditions to more than 10^{-5} s^{-1} , showed a strain-rate dependence of the yield strength.

INTERACTION BETWEEN DISLOCATIONS AND GRAIN BOUNDARIES IN NANOSCALE SAMPLES

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Abstract :

The strength of metallic materials depends on the length scale associated with the material. This length scale is in the form of an external length scale such as specimen size or in the form of an internal length scale such as grain size. The effect of grain boundary in ultra fine grained (UFG) and nanocrystalline (NC) metals is of interest. The newly developed method, compression test of micropillars, was used to investigate both the specimen size effect and the grain boundary effect on the strength of metals. Single and bicrystal micropillars with different crystallographic orientations were fabricated by focused ion beam system and the compression test was performed in a nanoindenter utilizing a flat punch tip. As a new approach in order to observe the micropillars in their intermediate deformation conditions, the compression test was performed stepwise. Between each step these micropillars were imaged in a scanning electron microscope. By comparison of yield strength of single and bicrystal pillars, an increase of the yield strength by decreasing the pillar size has been observed. Bicrystal pillars also show a higher strength compared to single crystals. The results show that the grain boundary has an obvious effect on the plastic behaviour in the tested Ni Bicrystals and pop-in behaviours are influenced by the size and the orientation of different adjoining crystals of the pillars. This effect cannot be simply explained for the smaller grain size in the bicrystals.

MECHANICAL PROPERTIES OF NANOCRYSTALLINE AND ULTRAFINE IRON AND CARBON STEELS

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Abstract :

Samples of nanostructured iron (0.05 C wt%) and steels with a carbon content of 0.3% and 0.6% wt. have been obtained by warm consolidation from mechanically milled powders. The samples have been consolidated at temperatures between 400° and 500°C. In order to obtain samples within the ultrafine regime, some of them were subsequently heat treated at temperatures between 650°C and 775°C.

The evolution of the microstructure with the heat treatment has been studied by SEM and TEM techniques. The presence of carbon atoms dissolved in the mechanically milled ferrite prevented the grain from excessive growth in the 0.3% and 0.6% carbon steels. For the 0.05% C iron samples, the minor concentration of carbon atoms resulted in a difficulty to control the grain growth and this led to bimodal grain size distributions.

The stress-strain response has been studied by means of compression and tensile tests. The nanostructured materials showed poor results in terms of ductility together with a high strength. Nevertheless, within the ultrafine regime strain hardening was easily observed in the compression tests. In the tensile test, the ductility was increased as the carbon content decreased from 0.6% to 0.05%. In the latter material, an interesting good combination of tensile strength and ductility has been found.

SURFACE PATTERNING OF AUSTENITIC STAINLESS STEEL BY PLASMA ASSISTED DIFFUSION TREATMENTS

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Abstract :

The new concept of surface texturing or surface patterning on austenitic stainless steel by plasma assisted diffusion treatment is presented in this communication. It allows the creation of uniform micro or nano relief with regularly shaped asperities or depressions. Plasma assisted diffusion treatments are based on the diffusion of nitrogen and/or carbon in a metallic material at moderate to elevated temperatures. Below 420°C, a plasma assisted nitriding treatment of austenitic stainless steel produces a peculiar phase usually called expanded austenite, S phase, ϵ phase or δ phase. Expanded austenite is a metastable nitrogen supersaturated solid solution with a disordered fcc structure and a distorted lattice. This phase is hard, wear resistant and a paramagnetic to ferromagnetic transition takes place for nitrogen content higher than 14 at%. Other particular features of the expanded austenite are high nitrogen content (from 10 to 35 at%) and high compressive residual stress in the nitrided layer. From mechanical consideration, it is shown that the only possible deformation occurs in the direction perpendicular to the surface. Such an expansion of the layer from the initial surface of the substrate to the gas phase is used here for surface patterning of stainless steel parts. The surface patterning is performed by using masks (TEM grid of different mesh side). High amounts of nitrogen (30 at%) are introduced in stainless steel over 2 to 3 μm from the initial surface. The lattice expansion (around 10 %) of the whole nitrided layer produces selective extrusion of square dots (200 nm to 1000 nm from the initial surface).

For patterning, we use a multi-dipolar plasma, arranged in a two-dimensional array, belonging to the new generation of low-pressure, high-density plasma sources providing independent substrate biasing and independent ion flux and ion energy control. The resulting plasma diffuses towards the substrate-holder that can be independently heated and/or biased. It is thus possible to carefully control the sputtering of the sample surface. The engineered surfaces produced by patterning obtained by plasma assisted diffusion treatments have new functional properties associated to potential interesting industrial applications.

Organic materials

ANALYSIS OF THE PET P SEMICRYSTALLINE MORPHOLOGY AND THERMOMECHANICAL PROPERTIES FOR LONG TERM PHYSICAL AGING RESEARCH

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Abstract :

This investigation aims at studying the mechanical and microscopic properties of PET P (polyethylene terephthalate) used for insulators in High Voltage Gas Insulated Substation before a long term physical ageing. For this reason, a basic study to understand the PET behavior in a reference state (before any thermal ageing) is definitely necessary. In a first step, the study focuses on revealing more detailed information about the PET morphology. For this purpose, PET samples have been subjected to special schemes of crystallization. In fact, after being quenched (amorphous state) from the semicrystalline state, the samples were annealed at different temperatures for several times. This controlled crystallization has shown a classical increase of the long period and the crystallinity ratio. The specimens have been characterized by different techniques, namely, differential scanning calorimetry (DSC), mechanical tests, and wide and small angles X scattering (WAXS, SAXS, respectively). The second step deals with the mechanical properties. We have studied closely the thermal effects below and above the glass transition (T_g), then the tensile rate effects during true stress-true strain tests. The mechanical measurements have shown that the Young modulus and the yield stress decrease when the temperature is increased. However, the material is slightly sensitive to the tensile rate below T_g or even unaffected above T_g .

THERMO-OXIDATIVE AND HYDROTHERMAL AGEING OF EPOXY-DICYANDIAMIDE ADHESIVE IN BONDED STAINLESS STEEL JOINTS

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Abstract :

The ageing behaviour of stainless steel joints bonded with hot-curing adhesives is crucial for their reliability and durability in engineering applications. After accelerated artificial ageing regimes, short-term mechanical tests are broadly applied in industry. They shall monitor the in-service long-term behaviour appropriately for predicting the life time of the adhesive joints. In that course of characterisation, chemical changes in the adhesive are widely disregarded. Hence, neither the true causes for the decreasing performance of the joint nor their relevance for application can be considered. On this reason, lap shear samples of the stainless steel alloy 1.4376 (improved energy dissipation capability due to N-content, increased Mn- and reduced Ni-concentration) are bonded with an epoxy adhesive (diglycidyl ether of bisphenol A and dicyandiamide, mass ratio of 100 : 6.7) cured, aged under moderate thermo-oxidative (60 °C, dried air) or hydrothermal (60 °C, distilled water) conditions, and tested by stress-strain analysis under shear load. On the fracture faces, chemical modifications of this adhesive due to ageing are detected by μ -ATR-FTIR-spectroscopy as function of ageing time and position in the adhesive joint. These data are correlated to the compound strength.

In contrast to the current practice in industry, this new approach is able to detect the chronological and local progression of the chemical modifications of epoxy adhesives in bonded stainless steel joints in consequence of whose accelerated artificial ageing. It is possible to relate these modifications to changes of the mechanical performance, too.

PC/PET REACTIVE BLENDS: FRACTURE ANALYSIS USING EWF APPROACH

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Abstract :

Polyethylene terephthalate (PET) recycling represents one of the most successful and common examples of polymer recycling due to its wide-spread use, particularly, in beverage industry. There has been considerable commercial interest in blends of PET with Bisphenol-A-Polycarbonate (PC) due to the possibility of interchange reaction to occur. This has opened a door to PET or PC recycling, as the product offers outstanding properties. However, few studies have been done to investigate the mechanical properties of PC/PET blends especially PC-rich ones focused in their fracture behavior. In this work, thermal, mechanical and fracture behaviour of sheets of PC/PET blends have been evaluated. Blends were prepared with and without catalyst based on Ca, Zn and Sm in a proportion of 0.4 % by weight of total amount of blend. According to FT-IR, DMTA and DSC results, Sm catalyst seems to be most effective in PC modification under the mixing/processing conditions used.

However, it shows the highest tendency to promote secondary reactions that degrade the system. Essential Work of Fracture analysis (EWF) has led to conclude that the addition of 20 %w/w of PET to PC with Zn catalyst offers the best balance on fracture parameters. It can be attributed to the degree of dispersion reached, interaction between phases and the crystalline nature of the PET droplets. According to fracture surface analysis by SEM, the amount of plastic work dissipated during deformation and fracture process is enhanced as a consequence of PET crystalline droplets cavitation that release local triaxiality in PC matrix.

CELLULOSE ACETATE GRAFT COPOLYMERS WITH NANO-STRUCTURED ARCHITECTURES FOR THE PURIFICATION OF BIO-FUELS BY A MEMBRANE SEPARATION PROCESS

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Abstract :

Cellulose acetate (CA) is a very good film-forming material for aqueous membrane applications. It has also been reported for the challenging separation of purely organic mixtures by pervaporation with outstanding selectivity for the purification of the ethyl-tertbutyl ether (ETBE) bio-fuel. Nevertheless, its flux was much too low to allow any further development for this application. Following a few works having shown the true potential of CA plasticizing for this separation [1, 2], CA-g-poly(methyl diethylene glycol methacrylate) copolymers were obtained by Atom Transfer Radical Polymerization (ATRP) by grafting from brominated macroinitiators. The copolymerization scaling-up up to ca. 5 g of copolymer samples was successfully achieved and two families of graft copolymers with nano-structured architectures were eventually obtained. The grafting from a first macroinitiator containing 6 initiator groups for 100 glucosidic cycles led to a first family of graft copolymers with a high number of poly(MDEGMA) short grafts. A second macroinitiator with 1 initiator group for 100 glucosidic cycles then enabled to obtain a second family of copolymers with almost the same graft weight fractions but with a low number of long grafts. This paper focuses on the sorption and pervaporation properties of both families of graft copolymers for the purification of the ETBE bio-fuel. As an example, the striking results displayed in Figure 1 showed that the copolymer properties did not depend only upon their highly permeable graft content but that their nano-structured architecture also played a key role in their separation behaviour.

**EFFECT OF REPEATED CONTACT ON ADHESION MEASUREMENTS
INVOLVING POLYDIMETHYLSILOXANE STRUCTURAL MATERIAL**

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Abstract :

During the last few years several research groups have focused on the fabrication of artificial gecko inspired adhesives. For mimicking these structures, different polymers are used as structure material, such as polydimethylsiloxanes (PDMS), polyurethanes (PU), and polypropylene (PP). While these polymers can be structured easily and used for artificial adhesion systems, the effects of repeated adhesion testing have never been investigated closely. In this paper we report on the effect of repeated adhesion measurements on the commercially available poly(dimethylsiloxane) polymer kit Sylgard 184 (Dow Corning). We show that the adhesion force decreases as a function of contact cycles. The rate of change and the final value of adhesion are found to depend on the details of the PDMS synthesis and structuring.

STUDIES OF NEW LOW MOLECULAR WEIGHT ORGANOGELS

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Abstract :

Low-molecular-weight organic gelling agents are currently subject of increasing attention, not only because of the numerous applications of gels, but also because understanding the detailed structural information of molecular assemblies is important for the rational design of those materials. Spectroscopic techniques such as nuclear magnetic resonance (NMR), infra-red (IR), fluorescence emission and circular dichroism (CD) are commonly employed with the aim at elucidating the detailed structure of supramolecular gels. A widely accepted mechanism of gelation is the spontaneous self-assembly of individual molecules into fibers and subsequent entanglement of these fibres into fibrous networks, including solvents in the interstices. The driving force for the self-assembly of molecules into fibres are non-covalent forces such as hydrogen bonding, π - π stacking, van der Waals interactions, solvophobic interaction etc. We described a new family of aminoacid or dipeptide type gelators obtained via an easy and inexpensive way. These novel gelling agents are very easily prepared from cheap starting materials and allowed many structural variations of the side chain. By taking advantage of the the presence of four points of diversity on our molecules, we underwent a rational study for a better understanding of the gelation phenomenon. We demonstrated that these systems are able to gelify organic solvents at very low concentration. In order to go deeply into the organogelation phenomenon, we point out the self-assembling behaviour of this kind of gelator which bears a fluorescent chromophore (a naphthalimide moiety) through IR, NMR, fluorescence spectroscopy. Highly oriented network structures were observed at gel state and disappeared in isotropic solution. The influence of the chirality and the nature of the lateral chain on the gelation properties will be discussed

Composite materials

CHARACTERIZATION OF DAMAGE EVOLUTION IN THERMO-MECHANICAL LOADING OF COMPOSITES MATERIALS

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Abstract :

In the experimental part, the displacement field on the edges of a [0, 554, -554]_s GF/EP laminate and [302,-602]_s GF/EP laminate specimens with multiple transverse cracks is visualized and analyzed on dependence of the applied mechanical load by using ESPI (Electronic Speckle Pattern Interferometry).

The different displacement profiles along the tensile-axis are drawn along the specimen edges at several distances from the mid-plane corresponding to the different plies. The measurements out-of-plane deformations are effected to determine and to compare the crack opening displacement (COD) with the crack face sliding displacement (CSD).

In the numerical part, the strong dependence of the average normalized (COD) on the relative stiffness and thickness of the surrounding layers, based on FE results, is described by a simple empirical expression for non-interactive cracks.

The FEM simulation results obtained by ABAQUS /CAE are compared with the general expressions for calculation of elastic constants of the damaged laminate developed by Lundmark and Varna [1]. The effect of crack interaction on the crack opening displacement (COD) is also discussed in this study. The effect on (COD) is described by the introduced "interaction function" which is determined using FE analysis.

MODELING MECHANICAL FATIGUE OF UD COMPOSITE : MULTIPLE FIBER BREAKS AND DEBOND GROWTH

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Abstract :

The objective of this paper is to analyze fiber/matrix debond crack growth in unidirectional (UD) composites during high stress cyclic tension-tension loading. When UD fiber reinforced polymer composites (CF/EP, GF/EP) are loaded in fiber direction in quasi-static or in a high stress cyclic tension-tension regime, multiple fiber breaks occur in random positions already during the first cycle. In cyclic loading with constant amplitude fibers are usually assumed not to experience fatigue. Therefore the damage evolution with increasing number of cycles is in form of development of interface cracks (debonds) growing along the fiber/matrix interface and thus fracture mechanics concepts (energy release rate) may be used for the damage evolution analysis. The sequence of events at strain levels below the fiber breaking limit may be different, for example, initiation of small matrix cracks between neighbouring fibers could be the first mode of damage, which would be followed by fiber/matrix debond crack propagation along fibers. However, for the studied CF/EP and GF/EP composites the energy release rate is much smaller for matrix crack initiated debond growth, besides energy release rate decays with the distance from the primary matrix crack.

Therefore only debonds initiated from fiber breaks are of interest if we consider high stress cyclic loading. First an analytical solution for the strain energy release rate in the selfsimilar debond propagation region is given. For short debonds, where the stress perturbation from the fiber break is interacting with the stress perturbation from the debond crack tip, FEM using virtual crack closure technique [1] is used to calculate the magnification of . The calculations are summarized in simple expressions and used in simulations of the debond growth in tension-tension fatigue according to Paris law expression [2].

EFFECT OF THERMAL AGING AND FATIGUE ON FAILURE RESISTANCE AEROSPACE COMPOSITE MATERIALS

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Abstract :

The increasing needs of aeronautical applications have turned interest to composite materials. This is due to high mechanical and thermal properties, at a lower weight, longer lifetime and reusability of composites. Some of the issues that these materials face are caused by exposure to high temperatures, large temperature variations (-50 to +40 oC) and mechanical fatigue due to pressure difference and vibrations of various structural components during a flight. The prementioned are main damage sources, in terms of cracks initiation and propagation and they are responsible for the material's degradation¹. In the present paper the effect of thermal aging and thermal fatigue on mechanical performance of carbon fibre laminate composites in tensile quasi-static loading and in mechanical fatigue have been studied. The main goal is to quantify the effect of the above degradation sources in terms of reduced resistance to matrix crack initiation and propagation in laminated structures. Multiple matrix cracks, called also intralaminar cracks, are observable in layer with off-axis orientation with respect to the main loading direction. These cracks usually initiate in weakest positions or in stress concentration areas, they cover the whole layer thickness and grow parallel to fibers in the layer. The statistical transverse failure properties in terms of Weibull distribution in the layer can be obtained from experimental data on cross-ply laminates expressing the linear crack density as a function of the applied strain which is translated to the thermo-mechanical stress in the layer. Weibull parameters are compared for the "healthy" untreated composite and the material after certain thermal treatment. The thermal aging consisted of exposing a sample at temperature close to the curing temperature for a time interval. Then mechanical loading and unloading in steps with increasing maximum strain is performed, measuring the number of cracks after every step. The mechanical fatigue consisted of a cyclic tension-tension load application and the relation between the number of cycles and the number of formed cracks was investigated for cross-ply carbon fibre laminates. Efforts were made to understand if the multiple damage in tests is initiation or propagation governed. In the first case the stress to initiate defect large enough to propagate requires high stress which exceeds the applied stress level needed for crack propagation. The crack propagation is unstable. If the initiation stress is low the cracking is propagation governed and can be stable as well as unstable. Anyway, in this case the fracture mechanics can be used

to describe observations and one of the expected phenomena is dependence of the multiple cracking characteristics on the cracked ply thickness. Thermal treatment can differently affect the degradation of initiation and propagation resistance characteristics. An additional study to separate the initiation from propagation was based on degradation of one edge of the specimen. To introduce defects on the edge the edge of the cross-ply specimen was immersed in liquid nitrogen for a very short time and then placed in the furnace. After repeating this procedure several times the same mechanical loading routine as before was used to compare the differences in cracking.

PREDICTION OF CRACK ONSET STRAIN IN COMPOSITE LAMINATES AT MIXED MODE CRACKING

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Abstract :

Failure process of continuous fiber reinforced composite laminates in tension usually starts with appearance of intralaminar cracks. In composite laminates with complex lay-ups and/or under combined loading, intralaminar cracks may develop in plies with different reinforcement directions. An obligatory part of mixed mode cracking models is the criterion of cracking, usually formulated in terms of energy release rates and their critical values for the specific material. Intralaminar fracture toughness of unidirectionally reinforced glass/epoxy composite was experimentally determined at several Mode I and Mode II ratios.

It is found that the crack propagation criterion, linear in terms of the energy release rates, reasonably well approximates the test results. The determined mixed mode cracking criterion was applied to predict intralaminar crack onset in [02/902]_s cross-ply glass/epoxy composite under off-axis tensile loading. The predicted crack onset strain values agree with test results at small loading angles (on-axis and 15° off-axis loading), but underestimates crack onset at larger (30° and 45°) loading angles. The discrepancy is likely to be caused by the deviation of linearity in laminate response before cracking onset in these laminates, related to non-linear shear characteristics of unidirectional plies.

MECHANICAL MODELLING OF SANDWICH COMPOSITES

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Abstract :

Due to their outstanding mechanical properties sandwich composites are of high interest in science and industry with special applications in the field of light weight constructions. The present contribution is up to the continuum mechanical modelling of sandwich structures consisting of two slight aluminium top panels and a polymer core layer. Whereas the material properties of the top panels can be described in an adequate way using a standard elasto-plastic constitutive model, further investigations have to be carried out concerning the viscoelastic core material. In the first part of the talk, we will give a brief review on the continuum mechanical treatment of viscoelasticity in general. Special attention will be spent to the understanding of the underlying rheological models. Thus, the effect of viscoelasticity can be split into the model of one spring element, representing the time independent basic elasticity of the material, and a parallel set of one or more Maxwell elements describing the viscous material response, i. e. the dependency on the strain rate. In the second part of the talk, some numerical examples will be given, on the one hand investigating the core material, on the other hand the composite structure.

BIODEGRADABLE COMPOSITE SCAFFOLDS FOR TISSUE ENGINEERING

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Abstract :

The most common and well studied family of bioactive calcium phosphate glasses is that containing Si as main component. Their osteoconductive properties have been well proved. These glasses have been widely investigated for their implantation on their own as granules, or as the reinforcing phase in composites. However, another family of glasses, Si-free, with controlled solubility and a predictable dissolution rate has also been developed more recently. Different formulations of these latter phosphate glasses have been developed and investigated. More specifically, glasses in the system P_2O_5 -CaO-Na 2 O-TiO $_2$ have been successfully developed by the research group at UPC. Their physicochemical, morphologic, and structural evolution has been analyzed during in vitro degradation in SBF (simulated body fluid) and the results show that the addition of TiO $_2$ into the glass system enhances both the elastic modulus and their chemical durability. The incorporation of small radius and highly charged ions contributes to the improvement of their chemical stability. The solubility control represents a great advantage in comparison with CaP crystalline ceramic materials used in orthopaedic applications. More recently, it has been shown that the soluble ions released upon degradation induce endothelial cells precursors to form vessels.

Moreover, these glasses have been also used as the reinforcing phase in PLA matrix composites. Scaffolds prepared from this material are totally degradable and seem to induce bone formation by promoting angiogenesis. In this sense, such composites could play a leading role in bone tissue engineering.

POSTERS

MICROSTRUCTURE AND THERMOMECHANICAL BEHAVIOUR OF SiC-BASED REFRACTORIES

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Abstract :

Refractory materials are commonly used in waste incinerator plants. They compose part of the wall covering the combustion chamber and protect metallic cooling tubes. Refractories are subjected to severe thermal gradients, to corrosion and to high temperature levels. They also collect part of the thermal energy liberated during the waste combustion in order to allow electrical energy production. Such materials must couple a high thermal conductivity and high thermomechanical properties to cope with running solicitations.

Nitride-bonded silicon carbides (NBSC) are porous materials with high thermal and mechanical properties. Microstructures and thermomechanical properties are investigated in order to understand their behaviour in incinerator plants. Specific properties come from their processing route. A mixture of SiC grains and Si powder is pressed to obtain the tile shape. Tiles are then sintered in a nitrogen-rich atmosphere. The resulting microstructure deals with SiC grains bonded with silicon nitride phases. Such bonding phases are characterised by a high complexity degree. Microstructural properties of two NBSC grades are studied. They deal with crystalline phase identification (by X-ray diffraction), phase morphology (by SEM) and porosity characterisation (by mercury porosimetry).

As the material is used at high temperature in oxidizing atmospheres, oxidation occurs.

Long-term oxidation effects are investigated. Phase and morphological changes but also specific area and porosity evolutions with temperature are considered. Kinetic aspects of oxidation phenomena are taken into account too.

In the 20°C-1200°C temperature range, the two NBSC grades exhibit a brittle behaviour.

Thermomechanical properties are studied and compared with oxide bonded SiC refractories and with SiC-based castables ones. Effect of long-term oxidation on the thermomechanical behaviour is investigated too.

FUNCTIONAL FILLERS IN HOT CURING EPOXIES FOR PROLONGED SHELF LIFE AND REDUCED CURING TEMPERATURE

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Abstract :

Epoxy formulations based on the multi-functional amine hardener dicyandiamide (Dicy) regularly contain a free accelerator for reducing the curing temperature and the time needed to complete the network formation. Unfortunately, all accelerators reduce the shelf life of these adhesives at 25 °C.

In order to solve this problem, accelerator-loaded zeolites fillers were developed, optimised with respect to host-guest interactions and characterised by Fraunhofer IFAM (Bremen, Germany) with regard to the release and curing behaviour in epoxy adhesive formulations.

They are added to an epoxy adhesive (EP = diglycidylether of bisphenol A and dicyandiamide (Dicy), mass ratio 100 : 6.7), stored at 25 °C in regular air or cured (heated with $\beta = 10 \text{ K / min}$ to 170 °C subsequent isothermal curing for 45 min).

That shelf life and curing behaviour are investigated by FTIR-spectroscopy and modulated DSC. Compared to the EP containing free accelerator, the zeolite-filled EP possesses a threefold time of shelf life at 25 °C due to the immobilisation of the accelerator in the pores of the zeolites. While the free accelerator acts steadily during heating, it is shown that the loaded zeolite releases the accelerator at about 95 °C. Surprisingly, the released accelerator is not only involved in the chemical formation of the epoxy network but it accelerates the dissolution of Dicy considerably. As the result, network formation at 170 °C finishes by about 30 min faster than for conventional adhesive formulations and the starting temperature for curing could be reduced to 140 °C.

MOLECULAR DYNAMICS OF AN EPOXY-DICYANDIAMIDE ADHESIVE: CURING AND CHEMICAL VITRIFICATION

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Abstract :

Epoxy formulations based on the multi-functional amine hardener dicyandiamide (Dicy) regularly contain a free accelerator for reducing the curing temperature and the time needed to complete the network formation. However, the influence of these accelerators on the network formation is not well understood yet. Besides, the dielectric spectroscopy (DES) has never been used before to monitor the network formation of such hot curing epoxy systems.

In this presentation, the isothermal curing of an accelerated epoxy adhesive (EP) is studied by real-time broadband dielectric spectroscopy at the curing temperature of 170 °C after heating with 8 K / min. The epoxy system consists of diglycidylether of bisphenol A (DGEBA) and dicyandiamide (Dicy) as the monomers and of 1-methylimidazole (1-MI) as the accelerator. The composition is chosen as 100 : 6.7 : 0.54 by mass.

Short-time frequency sweeps are performed for observing the network formation. In view of the high curing rate of the accelerated EP during the heating and at curing temperature two sweeps are considered for two sets of uncured samples: Sweep A with 1 Hz, 10 Hz, and 100 Hz and sweep B with 103 Hz, 104 Hz, 105 Hz, and 106 Hz. Due to the short measurement time, each frequency sweep represents an isostructural state while ensuring a high resolution. As the results, the dynamic glass transition (α -relaxation) and the β -relaxation are determined as functions of the curing state. This new approach allows evaluating the chemical vitrification as systematic decrease of the dielectric permittivity during the curing.

**RELAXATION PHENOMENA IN A $Zr_{44}Ti_{11}Ni_{10}Cu_{10}Be_{25}$ BMG ALLOY AS INVESTIGATED
WITH DILATOMETRIC AND CALORIMETRIC METHODS**

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We report on the relaxation kinetics of the $Zr_{44}Ti_{11}Ni_{10}Cu_{10}Be_{25}$ (Vitreloy 1b) alloy as measured with dilatometric and calorimetric methods. Using Differential Scanning Calorimetry (DSC) enthalpy is relaxed in the vicinity of the glass transition and the differences in enthalpy recovery between the differently relaxed samples are measured. A dilatometric method was optimized using Thermal Mechanical Analysis (TMA) and isothermal free volume relaxation in the vicinity of the glass transition is measured. The changes in enthalpy and average free volume are compared and found to be in good agreement with each other, as well as to similar research in the field. The free volume is furthermore found to relax with a stretched exponential behavior and is fitted best with a Kohlrausch-Williams-Watts (KWW) function with beta-values approaching unity close to the glass transition.

MICROSTRUCTURE ANISOTROPY IN POLYOLEFIN FLEXIBLE FOAMS

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Abstract :

The use of polyolefin flexible foams of typical thicknesses between 0.5 and 4 mm, which are produced by a combined process of extrusion and physical foaming, is nowadays quite widespread along the packaging sector. Their closed-cell structure, alongside the high flexibility, allows them to show good energy absorption properties under low loading conditions. Although the compressive response of these materials is well known, their inner microstructure, developed during processing, induce a high anisotropy that is responsible for their orientation-dependent tensile and fracture behaviours.

In this work, different polyolefin-based foams with densities ranging from 25 to 40 kg/m³ and thicknesses between 1 and 3 mm were studied. The induced microstructure anisotropy was characterized by micro-Raman. With this technique, the relative orientations of both crystalline and amorphous phases in the foam's base polymer could be determined and thus related to their mechanical properties measured in the different directions.

EFFECT OF THERMAL TREATMENTS ON THE WEAR BEHAVIOUR OF DUPLEX STAINLESS STEELS

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Abstract :

Duplex stainless steels (DSS) are a family of steels characterized by a two-phase microstructure with similar percentages of ferrite (α) and austenite (β). Their increased use on marine and petrochemical industries in the last decades is a result of their attractive combination of mechanical properties and corrosion resistance.

Nevertheless, an inappropriate heat treatment can induce the precipitation of secondary phases which directly affect their performance. On the other hand, there are few works dealing with the influence of heat treatments on the wear behaviour of DSS. Therefore, this paper aims to determine the wear kinetics and to investigate the sliding wear mechanisms developed as a function of thermal treatment conditions. Thus, samples were treated in a range of temperatures between 850 and 975 °C before performing sliding wear tests.

Microstructural observations showed the presence of sigma phase for all the studied conditions. Wear testing was carried out by ball on disk technique at constant sliding rate and different sliding distances. Two methodology techniques were used to calculate the wear volume: weight loss and area measurement using a simplified contact model. Results showed that the hardness increase due to the presence of sigma phase plays an important role on the wear behaviour of DSS. Wear rates decreased when increasing the percentage of sigma phase on the microstructure.

THERMAL STABILITY IN AIR OF SURFACE NITRIDED 3Y-TZP

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Abstract :

3Y-TZP ceramics had been widely used for the fabrication of femoral heads, because of their good combination of properties like high bending strength, moderate toughness, excellent biocompatibility and high wear resistance. Nevertheless, low temperature degradation, LTD, have represented an important drawback due to dramatic loss of mechanical properties. Among many different methods that have been applied to avoid LTD, nitridation is a promising technique because it does not induce changes in mechanical properties, if the process is carefully controlled. On the other hand, thermal stability in air at high temperature of nitrided Y-TZP has not been studied before. In this work, 3Y-TZP has been nitrided by contact with ZrN at 1400°C for 1 hour, in N₂ atmosphere. Then the material was heat treated in air at 400, 600 and 800°C for 8 hours. When treated at 400 and 600°C there was no change in mechanical properties and surface morphology. However, with the treatment at 800°C, surface cracks were formed due to nitrogen leakage, and fractured cross sections showed high intergranular decohesion in the first 50 microns of depth. Surface hardness did not change, but fracture toughness had an important drop, as the samples broke while being indented. This phenomenon is explained based on surface blistering by nitrogen.

EMBRITTLEMENT MECHANISMS AT HIGH TEMPERATURES FOR A C-MN STEEL WITH HIGH RESIDUALS CONTENT

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Abstract :

Transverse cracking in the surface of semi-finished products is a problem related to the continuous casting steelmaking route. It is believed that cracks form during the unbending operation which is carried out at temperatures for which most steels exhibit poor ductility. This behaviour is related to the appearance of different embrittlement mechanisms which mainly depend on the thermomechanical conditions and chemical composition of the steel.

In order to characterize the embrittlement mechanisms associated with the presence of residual elements, a C-Mn steel produced from scrap recycling was evaluated in the as-cast and as-rolled conditions. Hot tensile tests were performed to assess the hot ductility and characterize the embrittlement mechanisms of the aforementioned steel. In addition to the initial condition of the material, i.e. as-cast vs. as-rolled, the effect of the thermal cycle prior to deformation was also evaluated. The %RA (reduction area) vs. temperature curves showed ductility troughs starting in the austenite single phase region and extending until the lowest testing temperature. However, the ductility drop took place at higher temperatures for the as-cast condition than for the as-rolled one. The fractographic analysis of the samples showed different embrittlement mechanisms depending on the thermal cycle. In-situ melted samples showed interdendritic fracture associated with the microsegregation that took place during solidification. When samples were reheated at low temperatures, i.e. 1100°C, interdendritic features were still apparent because the reheating temperature was not high enough to homogenize the composition. On the other hand, high reheating temperatures, i.e. 1330°C, gave rise to intergranular embrittlement probably due to the intergranular segregation of the residual elements and/or impurities

CONTACT DAMAGE ALUMINA/MULLITE COMOSITES

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Abstract :

Alumina based ceramics are increasingly being used in many applications such as sliding components because of its wear resistance; however the inherent brittleness limits their use mainly in structural applications. Small mullite additions on alumina have been demonstrated to enhance its mechanical properties as well as its wear resistance.

Strengthening of grain boundaries and the improvement of the mechanical properties were proposed mechanisms for the higher wear resistance. In our work, Al₂O₃/5vol% mullite and Al₂O₃/10vol% mullite composites with different sintering conditions, different grain sizes, and consequently different microstructures were mechanically characterized. Hertzian contact tests on samples under different loads have been performed. SEM, AFM,

Interferometry and Confocal Microscopy have been used to characterize the surface and indepth damage on the samples. Contact behavior of materials was analyzed on the basis of material properties and microstructure. Materials with 5vol% of mullite and synthesized by reaction sintering process have exhibited the best contact damage response. Finally, a cross section of an indented sample was obtained by using FIB in order to analyze the quasiplastic region.

FATIGUE SUSCEPTIBILITY UNDER CONTACT LOADING OF CERAMIC THIN FILMS

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Abstract :

The tribomechanical response of ceramic coatings has been extensively documented in the literature. However, most of this work has been mainly conducted on the basis of indentation, scratch or well-established tribological (e.g. pin-on-disc or abrasive wheel) testing techniques. On the other hand, knowledge on their behavior under repetitive contact loading is rather scarce [1-4]. Considering that there is an increasing usage of ceramics coatings in applications where contact loads of cyclic nature between curved surfaces are common, e.g. cold forming tools and machine components; such information on both mechanical response and damage mechanisms involved during contact fatigue of coated systems is required if they want to be used effectively. In this investigation this is approached by assessing the contact fatigue behavior of two different PVD ceramic coatings deposited on a fine-grained hardmetal by means of spherical indentation testing techniques at both micro- and nano- scales. The use of a cemented carbide as a substrate is two-fold. On one hand, as it was referred above, it implies the study of a coated system of extended industrial application. On the other hand, such a stiff, hard and brittle substrate favors damage evolution from early circular cracks at the coating into direct substrate cracking, without any intermediate interfacial delamination stage [5]. Within this context, the intrinsic contact fatigue response of the studied coatings becomes even more relevant because early cohesive failure of the film may then be defined as the critical damage event for these coated systems. Spherical indentation tests at the micro-level indicate that both studied thin films: a TiN monolayer and a DLC-like multilayer, exhibit different susceptibility to fatigue degradation in terms of emergence of circular cracks at the coating surface. Such a distinct behavior is speculated to come from the different crystalline/amorphous nature of the investigated coatings [6]. Following these ideas, their intrinsic fatigue behavior and implicit deformation and failure mechanisms are then investigated by nanoindentation (both monotonic and cyclic) and advanced microscopic techniques (AFM and FIB). The variable fatigue sensitivity for the studied coatings is finally discussed in terms of the changes on mechanical response and damage evolution observed as applied contact stress and number of cycles increase for each ceramic thin film.

ENHANCING HYDROTHERMAL RESISTANCE OF 3Y-TZP USING CERIA DIFFUSION

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Abstract :

There has been interest in the last years to develop methods for improving the hydrothermal resistance of zirconia tetragonal polycrystals stabilized with 3 % molar of yttria (3Y-TZP). It is known that adding ceria (CeO_2) to the composition makes the tetragonal phase more stable under hydrothermal degradation; however the bending resistance of Ce-TZP is typically smaller than for Y-TZP. In the present work, different ceria diffusion treatments were studied by means of calcining 3Y-TZP with compacted CeO_2 powder at temperatures near 1400 °C for different periods of up to 10 hours. The objective was to optimize the treatment conditions while increasing the resistance to degradation. It is shown that during these treatments there is diffusion of cerium in a relatively shallow layer of about 10 μm in depth. Some of the considered treatments effectively prevented tetragonal to monoclinic transformation after exposure to water vapour in autoclave at 131°C during 30 hours. The observed macroscopic fracture toughness, Vickers hardness and Young modulus of the treated specimens showed no significant deviation as compared to the non-treated original material: 5 MPa $\text{m}^{1/2}$, 12 GPa and 245 GPa, respectively. Surface measurements by nanoindentation with penetration depths of about 2 μm corroborated that hardness and elastic modulus did not change significantly in this shallow affected layer. However, microstructural changes took place in the surface since the grain size increased with the time of treatment at 1400 °C. The largest grain sizes, between 1-2 μm , were observed for the specimens treated during 10 hours at 1400 °C. At this temperature a time of treatment of 5 hours already was sufficient to prevent transformation. The treatment at 1500 °C during 1 hour also prevented t-m transformation while at the same time increasing only slightly the average grain size to a minimum value of 0.44 μm as compared to other treatments.

Therefore surface treatment with CeO_2 is an effective method to increase resistance to hydrothermal degradation in 3Y-TZP polycrystals without reducing their excellent mechanical properties.

NEW SILICON-DOPED CALCIUM PHOSPHATE CEMENT FOR BONE REGENERATION APPLICATIONS

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Abstract :

Introduction α -tricalcium phosphate (α -TCP) has been widely used as powder reactant for the preparation of calcium phosphate cements for bone regeneration. Nevertheless, this crystallographic form is only stable above the β - α transition temperature, and it is necessary to perform a quenching to obtain it in a metastable form. It has long been known that silicon is an alphasen element, and more recently, wet chemical methods were presented to obtain a Si-stabilised α -TCP with improved bioactivity. In this work, the preparation of calcium phosphate cements doped with silicon is proposed, using a novel method for the fabrication of Si-stabilized TCP where no quenching is required. Materials and Methods Highly pure hydroxyapatite (HA) and an aqueous suspension of nanometric SiO_2 (SO) were mixed with water and milled to ensure the intimate mixture of the reactants.

The resulting paste was dried, sintered and slowly cooled down in the furnace. The effect of silicon content was evaluated and the optimal reactant ratio to prepare Si- α -TCP was selected. The calcium phosphate cement was prepared by mixing the Si- α -TCP powder with water; cylindrical moulds were filled with the paste and immersed in Ringer's solution.

Physical and chemical characterization was performed at different immersion times. Results and Discussion α -TCP was obtained only when silicon was incorporated and the amount of this phase increased with SO content. Silicon did not decrease the β - α transition temperature, but it did stabilize the α -phase even when no quenching was applied. Silicon doped cements fulfilled basic clinical requirements and completely transformed into a calcium deficient hydroxyapatite. The cements showed the same microstructure and mechanical behavior than those obtained with non-doped α -TCP. The effect of silicon incorporation on the biological performance is currently under study.

ESSENTIAL WORK OF EVOH COMPOSITES

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Abstract :

Ethylene-vinyl alcohol (EVOH) copolymers belong to random semicrystalline materials with excellent gas-barrier properties. In recent years, the interest from food packaging industry in using EVOH copolymers, in film form, has increased due to important specifications in terms of gas, aroma and flavour permeation. In order to improve the EVOH film properties, several technological alternatives like multilayer structures; blending; and more recently, EVOH/OMMT composites have been used. The latter, is commonly produced by melt intercalation method. The aim of this study is to prepare EVOH/OMMT composites and analyze the influence of the clay content on mechanical properties and its effect on fracture behaviour by applying the essential work of fracture (EWF) method in the two main extrusion directions: melt flow direction (MD) and transverse direction (TD). The results indicated good EVOH/OMMT compatibility as well as the influence of the clay particles arrangement into the fracture behaviour. Besides, the OMMT particles inside the films were disposed with a preferential orientation along the MD axis. As a result, in MD, the OMMT is acting as positive reinforcement for the EVOH matrix. Then, the presence of the clay particles promotes an increase on the w_e term in both MD and TD, due to the EVOH/OMMT interaction. Meanwhile, the specific plastic work, w_p , decreases with the presence of the OMMT particles in MD and TD, as a consequence of a hampering effect of the clay to the plastic flow.

BRITTLE-DUCTILE TRANSITION OF POLYLACTIC ACID (PLA) : EFFECT OF CLAY AND/OR OXO-BIO PRO-DEGRADING ADDITIVE

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Abstract :

In this work it has been studied the effect of addition of Organo-modified montmorillonite (Cloisite®30B, OMMT) and a Oxo-bio pro-degradation additive (Biomax®Strong 120, BIO) on the brittle-ductile transition of a Polylactic acid (NatureWorks®PLA Polymer 2002D, PLA) promoted by a thermal treatment (10 min. at 60°C followed by quench).

Composites were prepared by twin screw extrusion followed by a mould injection process. According to rheological, DMTA and DSC measurements addition of CLOISITE 30B (0.5 to 2.5 %w/w) seems to offer a double effect on composite. By one side a stiffener one (expected) and a plasticizer one (not expected) that counterbalance each other. This effect could be attributed to the organo-modification applied to the clay. On the other side, BIO (0.5 to 2.5 %w/w) acts as a plasticizer modified, promoting an increase on flexibility on the material. Hybrid composites (OMMT + BIO) show intermediate behaviour. Thermal treatment applied on pure PLA promotes an abrupt change on its mechanical behaviour characterized by a decrease in stiffness and an increase on ductility which promote a drastic change on fracture behaviour: from brittle to ductile. This is a consequence of a de-aging process that promotes an increase on molecular relaxation during mechanical. Addition of CLOISITE 30B restrict this effect while BIO additive seems to not change the trend observed on pure PLA.

EFFECT OF THE RESIDUAL STRESSES INDUCED BY GRINDING ON THE LOW TEMPERATURE DEGRADATION OF Y-TZP

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Abstract :

Tetragonal Zirconia Polycrystals doped with Ytria (Y-TZP) have been used as structural ceramics due to their excellent mechanical properties. Nevertheless the phenomenon of hydrothermal ageing has limited their use, since it reduces the strength with time and the surface is degraded by the tetragonal-monoclinic transformation accompanied by microcracking. In this work, it has been studied the influence of residual stresses induced by the grinding process on the hydrothermal degradation of Y-TZP. The distribution and quantification of monoclinic phase was studied by Raman spectroscopy in the cross section of ground samples, whereas the surface layer affected by the residual stresses was estimated by variation of crack lengths induced by Vickers indentation. The degradation treatments were performed in autoclave at 131°C and 0.2 MPa of pressure for different times. Results indicate that in ground samples the distribution of compression stresses presents a maximum at the points near the surface and decreases with depth until insignificant values at a depth of 9 μ m approximately. This result is correlated with Raman spectroscopy measurements, which show the tetragonal-monoclinic phase transformation produced by the grinding process, with a maximum concentration of monoclinic phase just below the surface of \sim 7% that decreases until 0% at a depths of about 7. Moreover, after degradation tests of ground samples no monoclinic phase can be detected by Raman spectroscopy and this suggests that the residual compression stresses produced by grinding increase the ageing resistance.

CONTACT DAMAGE IN ARTIFICIALLY AGED 3Y-TZP

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Abstract :

The behaviour under spherical indentation of hydrothermal degraded 3 % molar yttria stabilised zirconia (3Y-TZP) has been studied. The investigation is focused mainly on analyzing the response of the material under spherical indentation in terms of the transformed tetragonal to monoclinic degraded layer that appears after hydrothermal degradation. Three stages of damage have been analysed: a) the onset of permanent deformation on the surface; b) the development of sub-surface brittle or quasi plastic damage (surface chipping, ring crack formation and/or micro-cracking); c) final catastrophic failure under conditions of severe contact loads. A damage map is constructed to assess the resistance of 3Y-TZP for different degradation times in terms of diameter and depth of the indentation imprint, presence of pile - up and cracking. By this analysis critical loads are established for the appearance of surface damage and they are discussed in terms of the change in elastic properties of the surface degraded layer. Under critical loads for inducing ring cracks around the indent, a cross-sectional analysis also showed the presence of cone cracking.

INFLUENCE OF ELECTRIC FIELDS DURING THE FIELD ASSISTED SINTERING TECHNIQUE (FAST) AND DURING DRYING TECHNIQUES

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Abstract :

The present study investigates the influence of the electric field on the sintering behaviour of nanoscaled powders using a modified experimental setup of the Field Assisted Sintering Technique. The current path is restricted to the powder mould by electric insulation.

Thermal energy is supplied by a heating element positioned around the pressing equipment.

A design-of-experiments method was applied to investigate the influence of temperature, heating rate, pressure, holding time and electric field. The microstructure of the specimens was analysed by SEM. The results show that, besides temperature, which is the dominating factor, the heating rate and pressure have also an influence on the emerging microstructure.

There is no impact of the field on the microstructure of the insulating and semi-conducting powders under investigation. Furthermore, different experimental setups were built to examine the role of high electric fields on mass transport in dry pressed samples and during the drying process of dip coated films. Preliminary results show how the applied high voltage has an effect on the crack formation process in the dip coated films. This PhD-work was performed at the Saarland University, Germany at the institute of Prof. Dr. Rolf Clasen:

THERMAL CONDUCTIVITY OF NANO-POROUS THERMOELECTRIC MATERIAL

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Abstract :

We carried out molecular dynamics simulations of heat conduction in Si with a nano-hole to represent the nano-structure, in order to investigate the mechanism of the thermal conductivity reduction of nano-structured materials. Phonon dispersion curves are calculated by using the time-space 2D Fourier transform, and Phonon density of states is also calculated by the Fourier transform of a velocity correlation. We discuss the mechanism of the reduction of the thermal conductivity of nano-porous material on the atomic scale. We applied the thermal conductivity reduction of nano-porous material to thermoelectricity for enhancement of its performance. The thermal conductivity of a nanoporous bismuth telluride (Bi_2Te_3) thin film of nano-particles is measured by using the 3ω method with a temperature range from 100 K to 300 K. The $\text{Bi}_{0.4}\text{Te}_{3.0}\text{Sb}_{1.6}$ nano-particles with an average size of approximately 60 nm are fabricated by a beads-milling method. The nano-particle solution is prepared by mixing with toluene. A printing method is used to deposit the nanoparticle thin films onto a substrate, which is then sintered in hydrogen ambient. The measured thermal conductivities of nano-porous Bi_2Te_3 are from $0.09 \text{ W m}^{-1} \text{ K}^{-1}$ to $0.14 \text{ W/(m}\cdot\text{K)}$, these results are one order of magnitude lower the thermal conductivity values than that of bulk values. The bulk thermal conductivity increases with decreasing temperature, but the thermal conductivity of nano-porous Bi_2Te_3 thin film was constant at approximately $0.1 \text{ W/(m}\cdot\text{K)}$ at low temperature. It is considered that the phonon scattering at the nano-porous in the thin film is responsible for the measured reduction in thermal conductivity.

THEORETICAL AND NUMERICAL INVESTIGATION OF NANOINDENTATION OF POLYMER LAYERS

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Abstract :

Nanoindentation technique nowadays became a powerful and popular testing method because properties of very small specimen or thin films and even local mechanical properties of inhomogeneous materials can easily be studied. While nanoindentation got a reliable mean for the estimation of metal properties, properties of viscoelastic polymers cannot be determined by this method in a simple way. It is the goal of the present work to develop a robust method for the estimation of material parameters of viscoelastic models. To set up the stage, real experiments are replaced by finite element simulation of the indentation tests. The viscoelastic layer is modeled within a finite formulation allowing for the usual nonlinear behavior. In order to represent a certain spectrum of relaxation times, several Maxwell elements are connected in parallel. The indenter itself is assumed to be linear elastic and very stiff compared to the viscoelastic layer. The outcome of the model in form of force-displacement curves computed for different loading rates replaces real experimental data in the parameter identification process. For the numerical treatment the commercial finite element tool COMSOL Multiphysics® in combination with MATLAB® will be used allowing for an easy implementation of the required viscoelastic constitutive equations. The applied load history has to be optimised in order to allow for a separation of dissipative effects. It is proven how accurate the known material parameters can be reidentified from the numerical data quantificationally. Also variations of the geometry of the indenter tip as well as the friction between indenter and polymer layer are investigated.

QUANTITATIVE ESTIMATION OF STRUCTURE HOMOGENEITY OF MECHANICALLY ALLOYED DISPERSION-STRENGTHENED COMPOSITE MATERIALS

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Abstract :

It is important to achieve homogeneity of composite materials microstructure for high level of stability of its mechanical properties. In this work the technique of quantitative estimation on example of mechanically alloyed dispersion-strengthened composite material is offered.

Matrix of the composite material were a mixture of large chip particles of alloys Al-4%Cu-1.5%Mg and Al-12%Si-2%Cu-0.8%Fe. Composite material production included the combined treatment of the matrix mixture and SiC particles (grain size, 10 μm) in the amount of 20 vol.% in a planetary mill by quasi-cylindrical milling bodies in an argon atmosphere for 16–120 min. The compacted cylindrical samples 15 mm in diameter and 10 mm in height obtained by double-action compaction at a temperature 450 °C. Composite material structure homogeneity was carried out on compacted samples by examination of coefficient of variation (Cvar) of matrix phases and strengthening ceramic particles distribution. Research has shown that Cvar = 10 % corresponds to formation of homogeneous structure of mechanically alloyed composite materials. In the investigated composite material value of Cvar = 10 % is reached after 90 min of mechanical alloying.

ELABORATION OF NEW GRAFTED GLYCOPOLYMERS

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Abstract :

The objective of the present work is the synthesis of new amphiphilic, biocompatible and biodegradable grafted glycopolymers (dex-g-PChA), composed by a hydrophilic dextran (dex) biopolymer backbone grafted with hydrophobic poly(cholesteryl acrylate) synthetic blocks (PChA). These grafted glycopolymers may be particularly interesting as emulsifying agents or for the preparation of self-assembled and, fully biocompatible and biodegradable, drug delivery systems. A "grafting from" approach was performed to obtain these glycopolymers, using Atom Transfer Radical Polymerization (ATRP) technique for the polymerization of cholesteryl acrylate (ChA) with a modified dextran as macroinitiator.

Firstly, ATRP conditions were studied and optimized to obtain a well controlled homopolymerization of the ChA monomer. Homopolymerization of ChA was carried out in various solvent (Toluene, THF) using many type of ATRP ligands (e.g. PMDETA and n-Pr-PMI). The polymerization results showed a good control over the molecular weight and low polydispersity indexes. In parallel, organosoluble dextran based macroinitiators with a controlled number of ATRP-initiating groups along the chain were prepared by a two modification steps approach already published. Finally, desired grafted glycopolymers were obtained by ATRP polymerization of ChA with dextran based macroinitiator and a subsequent deprotection step of the dextran segment into hydrosoluble block. The prepared and modified macromolecules were carefully characterized by ¹H NMR, ²H NMR, and SEC analysis.

CORROSION RESISTANCE OF PEO COATINGS ON ALUMINIUM ALLOYS: EFFECT OF ELECTRICAL REGIME

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Abstract :

In many studies, PEO coatings are performed using complex electrical regime with negative and positive current density in pulsed mode without a clear justification of this choice. In this paper, aluminium alloys (2214 and 7175) were coated using PEO treatment in a KOH+Na₂SiO₃ containing electrolytic bath. The effect of different kinds of current shapes was compared in terms of structure and composition of the anodized films and corrosion performance of the coated material. Pulsed currents were used (with or without negative semi-period) with a Ceratronic generator as well as direct current. The characterization of the films was performed using X-Ray Diffraction and Scanning Electron Microscope equipped with an electron dispersion X-ray spectroscopy analyser. The corrosion resistance of the coated alloys was studied by classical electrochemical impedance spectroscopy, voltammetry and chronoamperometry. The first results show that the electrical regime influences the thickness, the roughness, the porosity and even the optical properties of the anodized layers. Pulsed currents provide a better corrosion resistance of the anodized aluminium alloys than direct current. However, pulsing the current in both positive and negative semi-period seems not to improve the protective action of the film.

**FORMATION OF BIOACTIVE COATINGS ON AZ91D MAGNESIUM ALLOY
BY PLASMA ELECTROLYTIC ANODIZING**

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Abstract :

Magnesium is a potential candidate as biodegradable orthopaedic implant due to its mechanical properties similar to natural bone. But its low corrosion resistance in the body

fluid is a limiting factor. By using electrolytic plasma (or micro-arc) anodizing process, 10 to 20 μm thick homogeneous protective coatings were formed on AZ91D magnesium alloys.

The anodic layers were grown with a simple electrical regime (at constant current density) in a classical alkaline electrolytic bath containing KOH, phosphate and fluoride anions. The effect of a small addition of CaO in the electrolytic solution was studied. The structure and composition of anodic films were investigated by X-Ray Diffraction and Scanning Electron Microscope equipped with an electron dispersion X-ray spectroscopy analyser. They contain crystallized MgO, crystallized MgAl_2O_4 , P and Ca whose contents depend on their concentrations in the electrolytic bath. The corrosion resistance of the coated materials was measured in simulated body fluid (SBF) at 37°C by voltammetry and impedance spectroscopy (EIS). Polarization curves and EIS results show a better resistance to general and pitting corrosion of treated alloys. A small amount (less than 1g.L⁻¹) of CaO added in the electrolytic bath improves furthermore the behaviour of anodized alloys in SBF. The bioactivity of untreated and treated AZ91D magnesium alloys was studied by long time immersion in SBF at 37°C. After 3 weeks, SEM-EDS characterization showed that all surfaces are covered by small particles containing mainly Ca and P (with a Ca/P ratio similar to the ratio in hydroxyapatite : 1.66-1.68). Treated surfaces are more covered than untreated ones. A small addition of CaO in the treatment bath enhances the covering rate.

LOCAL PERIODIC CHEMICAL DECOMPOSITION OF Cu₃N AND PdPtO THIN FILMS USING LASER INTERFERENCE IRRADIATION FOR TAILORED ELECTRICAL RESISTIVITY

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Abstract :

Microstructural evolutions are directly responsible for metallurgical effects on the physical, mechanical and also chemical behaviour of materials. A fortiori in the case of thin films, size effects may also lead to a more complex interpretation of the results and require more specific attention in the advanced microstructural design of metallic thin films as well as the detailed understanding of dependencies on the material response. Laser interference irradiation is used here to locally tune the microstructure of Cu₃N and PdPtO thin films deposited on glass substrates to induce a chemical decomposition which is responsible for the tailoring of the electrical resistivity. By interfering two laser beams of an high power pulse nanosecond Nd:YAG laser at the film surface, a precisely redesigning of the surface microstructure in terms of locally and periodic heating at the interference maxima positions could be achieved. This technique allows the local nucleation and crystallization of amorphous or nanocrystalline metallic thin films, combining nano- and microcrystalline regions ordered in periodic line-like arrangements in the composite architecture. Hereby a periodic chemical decomposition of the as-sputtered films resulting in a porous microstructure due to the emission of gaseous nitrogen and oxygen is reported.

Furthermore, XRD and EDX analyses have confirmed a further decomposition of the films in metallic compounds leading to a significant decrease of the electrical resistivity after the laser patterning

SOME METHODS OF INDUCING HIGH-COERCIVITY STATE IN EPITAXIAL MAGNETIC GARNET FILMS FOR THERMOMAGNETIC RECORDING

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Abstract :

One of the basic directions of advanced magneto-optics is the magnetooptical recording of the information, in particular, the thermomagnetic recording. The major requirement to materials-carriers is the presence of high coercivity. Therefore preparation of magnetooptical films with high figure of merit and great value of coercive force H_c is one of paramount problems of material science and technology of magnetic media. The epitaxial magnetic garnet films are rather promising materials for this application. The purpose of the present work was the development of methods of a high-coercivity state generation in magnetic garnet films for thermomagnetic recording. As objects of examination were used the following film compositions: $Y_3Fe_5O_{12}$, $(YSmLuCa)_3(FeGe)_5O_{12}$, $(YEuTmCa)_3(FeGe)_5O_{12}$, $(YBi)_3(FeGa)_5O_{12}$, $(YYbBi)_3(FeGa)_5O_{12}$, $(BiTm)_3(FeGa)_5O_{12}$ and $(YPrLuBi)_3(FeGa)_5O_{12}$. The ones were grown by a method of high-temperature liquid phase epitaxy from solution in melt on substrates $Gd_3Ga_5O_{12}$ and $(GdCa)_3(GaMgZr)_5O_{12}$ with crystallographic orientations $\langle 111 \rangle$ and $\langle 210 \rangle$. The following fluxes were used for epitaxy: $PbO-B_2O_3$, $PbO-Bi_2O_3-B_2O_3$ and $CaCO_3-Bi_2O_3-V_2O_5$. The coercive force was defined on a hysteresis curves and by method of oscillating field. The hysteresis curves were recorded with the magnetooptical hysteresigraph and with vibration magnetometer. The measured values H_c were: 80 A/m – 250 A/m for films of (Ca, Ge) system and 30 A/m – 720 A/m for films of Bi-containing garnets. On the basis of obtained results the effective ways of a high-coercivity state induction in epitaxial magnetic garnets were developed (among them the technological modes of growth, postepitaxial treatment, etc.):

- 1) Doping by Ca^{2+} ions;
- 2) Selection of a suitable relationship of ion concentrations (in particular, among Bi^{3+} and Yb^{3+} (Tm^{3+}) ions);
- 3) Postepitaxial treatment in a homopolar corona discharge.

**THE NATURE OF RADIATION COLOR CENTERS IN SINGLE CRYSTALS
Gd₂,6Ca_{0,4}Mg_{0,25}Zr_{0,65}Ga₄,1O₁₂**

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Abstract :

Owing to high transparency of (GdCa)₃(GaMgZr)₅O₁₂ single crystals in visible and near IR regions of spectrum, they are used as substrates of Bi-containing magnetic garnet films for applied magnetooptics. It is studied the nature of radiation color centers arising in single crystals of Gd₂,6Ca_{0,4}Mg_{0,25}Zr_{0,65}Ga₄,1O₁₂ under the influence of ultra-violet radiation, γ -quanta and fast electrons. The samples were plates with (111), (210), (110) and (100) crystallographic orientations. The treatment of samples included ultraviolet flare by the xenon lamp, γ -quanta irradiation (Co60 source, $E_\gamma = 1,25$ MeV) or irradiation by fast electrons ($E_e = 6$ MeV). It has been found that the spectrum of additional absorption (AA) in the irradiated crystals has complex system of bands from 17000-20000 cm^{-1} to 42000 cm^{-1} with maxima about 24000 cm^{-1} , 32000 cm^{-1} and 40000 cm^{-1} . Even at initial doses of irradiations in spectra there is wide band of AA with $\nu_{\text{max}} = 24000$ cm^{-1} , and initially transparent crystals get yellowy-brown colouring. Thermally stimulated polarisation spectrum of the irradiated samples consists of two groups of blocked peaks. A series of narrow intensive peaks is observed in the region of 300-390 K. In the region of 450-570 K the wide nonelementary absorption band is observed with a maximum at $T \sim 51-540$ K. Comparative studying of AA in crystals (GdCa)₃(GaMgZr)₅O₁₂, Gd₃Ga₅O₁₂, Gd₃Ga₅O₁₂:Ca²⁺, Gd₃Ga₅O₁₂:Mg²⁺ carried out. It is shown that formation yellowybrown colouring in gallium garnet crystals caused AA band with $\nu_{\text{max}} = 24000$ cm^{-1} , instead of band with $\nu_{\text{max}} = 29000$ cm^{-1} as was considered earlier. The complex structure of AA band with $\nu_{\text{max}} = 24000$ cm^{-1} is found out. The obtained results have allowed to develop the mode of rejection of substrates for devices with high value of magnetooptical figure of merit and the method of colouring of jeweller stones on the basis of garnet crystals.

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Abstract :

High power pulsed laser beams interact with a large number of materials and deliver a potent new process to modify surface microstructure, topography and chemistry. By using Laser Interference Metallurgy, coherent laser beams interact creating periodic interference patterns on irradiated surfaces. It has been proven that friction and wear properties of noble material thin films are dependent on micro structures induced by laser irradiation.

This new topographical way to reduce wear and friction has been applied to bulk copper and aluminum. Line-, dot- and cross-like patterns were generated in order to develop effective patterns and to understand the wear behavior of materials. Furthermore, the friction and wear properties of structured substrates were studied in both dry and lubricated conditions. The fluid transport in the laser structure was also studied. Different parameters (e.g. pattern periodicity, fluid viscosity) have been compared using two-dimensional finite element method (FEM) simulations.

**FORMATION OF INTERMETALLIC PHASES ON Ti/Al MULTILAYER SYSTEMS
BY LASER INTERFERENCE METALLURGY**

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Abstract :

The laser interference metallurgy method has been used to produce linear patterns with different periods in the micrometer regime. This technique allows the periodical modification of physical properties of surface layers or thin films. Basis of this processing is the reorganization of the microstructure caused by a laterally ordered heat treatment.

Especially the formation of intermetallics can optimize the mechanical properties such as hardness or Young's modulus. The interfering laser beams locally heat the sample at the intensity maxima. In this work, the time-dependent temperature distribution is simulated subject to the main processing parameters pattern period and laser fluence. By means of the simulation results it is possible to determine the maximum occurring temperature, temperature gradients and the span of time, during which the material is heated. Here, Ti/Al multilayered films with 300 nm total and 17 nm single layer thickness were irradiated by a Nd:YAG laser with a pulse width of 10 ns at a wavelength of 355 nm. The layer structure is completely dissolved at the intensity maxima and results in a completely altered microstructure. Besides aluminum and titanium grains, the formation of intermetallic phases can be shown using transmission electron microscopy. In addition, X-ray diffraction measurements were carried out in order to achieve integral data. The selected period ranged between 3 and 8 μm . It influences directly the newly formed microstructure.

Especially the grain size is sensitive to this respect, which is in agreement with the thermal simulation. Nanoindentation experiments show that the hardness increases at the intensity maxima due to the laser processing.

EXPANDING RESEARCH POSSIBILITIES IN MATERIALS SCIENCE BY CHARACTERIZATION WITH SEM/FIB DUAL BEAM SYSTEMS

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Abstract :

The development of high-performance materials is extremely correlated with the ability to carefully design their micro and nanostructure. This is only possible, if a precise characterization tool is available. Since new developments tend to the design of smaller structures in the nanometer range, the search for increasing resolution for characterization as well as for machining of samples is a constant task. The development of the focused ion beam (FIB) technique in the past decade facilitates completely new perspectives for target preparation, local structuring and micromachining as well as structural analysis using milling and imaging with a finely focused Ga ion beam. The combination of a FIB with a scanning electron microscope (SEM) adds additional possibilities of characterization. In the present contribution, different applications of the SEM/FIB Dual Beam System applied to different current research issues in materials science will be presented. Techniques like foil preparation for transmission electron microscope, cross sectioning, imaging with ions, scanning transmission electron microscopy and FIB-Tomography will be discussed. Applications include among other the characterization of nanowires and composite materials.

DYNAMICS OF PHOTO INDUCED PHASE TRANSITION IN THE SPIN- CROSSOVER COMPLEXES $\text{Fe}_x\text{Zn}_{1-x}(\text{phen})_2(\text{NCS})_2$, INFLUENCE OF METAL DILUTION

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Abstract :

Spin crossover (SC) compounds are among the most intriguing and fascinating functional molecular materials. They exhibit a High-Spin (HS) to Low Spin (LS) transition which can be triggered reversibly by temperature, pressure or light excitation [1]. Owing to their specific HS-LS bistability and photo-response properties, these materials may have potential use in the next generation of magneto-optical data storage devices [2]. For that purpose, recent advances have focused on the development of SC nanoparticles [3-4]. The dynamics of photo-induced phase switching is a crucial parameter which control the writing / erasing duration capabilities. We have undertaken a detailed study of the influence of metal dilution on the dynamics of photo-induced phase transition in the prominent spin-crossover compound $\text{Fe}(\text{phen})_2(\text{NCS})_2$. It has been shown that dilution controls the cooperativity and tunes the relaxation kinetics in $\text{Fe}_x\text{Zn}_{1-x}(\text{phen})_2(\text{NCS})_2$ [5]. Using x-ray powder diffractometry (XRD) under in-situ CW laser light excitation, we derive the kinetics of photo-conversion and relaxation as a function of temperature, laser power and metal dilution, non-linear effects resulting from a photo-induced instability are clearly pointed out. The results are interpreted in terms of domain nucleation and growth kinetics. In a second aspect, we have investigated the influence of the synthesis condition of $\text{Fe}(\text{phen})_2(\text{NCS})_2$, such as temperature, ultrasonic exposure and reagents concentration, for designing monodispersed nanoparticles.

DESIGN AND MANUFACTURE OF NICKEL / CARBON NANOTUBES COMPOSITES

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Abstract :

Nanocomposites reinforced with carbon nanotubes exhibit great promise, mainly because the CNTs are extremely stiff and excellent heat and electrical conductors. Novel nickel matrix composites reinforced with carbon nanotubes have been manufactured by powder metallurgy. After debundling the carbon nanotubes agglomerates, a mixture of nickel dendritic powder and multiwalled carbon nanotubes were compacted by cold pressing followed by a sintering process. A uniform dispersion of nanotubes in the metal matrix was obtained and a very good final density was reached. SEM observations and FIB cross sections of the composite are also presented.

CHARACTERIZATION OF RETENTION PROPERTIES OF CHARGE-TRAPPING MEMORY DEVICES AT LOW TEMPERATURES

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Abstract :

The ongoing scaling of the memory cell size has made the "charge trapping" memories one of most promising type of the nonvolatile semiconductor memories [1]. The present work deals with SONOS devices, which also belong to the charge-trapping memory type. It is based on a standard polysilicon field effect transistor but with a stacked gate insulator consisting of a silicon oxide-silicon nitride-silicon oxide sandwich. In this type of memories the information is stored in form of charges captured in silicon nitride or silicon oxynitride in spatially isolated deep level traps. Since namely the traps are responsible for the memory actions of SONOS devices, the extraction of their characteristics (density, energy and spatial distribution) is important for the prediction of retention properties of memory cells and also provides probability for their successful improvement. The present work focuses on the extraction of trap parameters (especially trap density distribution in energy) in silicon oxynitride using appropriate models. The studies were performed on MONOS (metal-oxideoxynitride- oxide-silicon) capacitors, which are the basic structures of technological memory cells. The trap density distribution can be extracted from the time dependence of the flat band voltage decay rate of the programmed memory cell [2]. This dependence can be derived from experimental retention characteristics – the flat band voltage shift with time. In the present work the retention characteristics were measured with a constant capacitance method. The advantage of the used technique lies in the constancy of the field conditions in tunnel oxide during the whole measuring time. The extraction of the trap distribution requires a deep understanding of discharge processes in the memory cell. Generally several mechanisms jointly contribute to the loss of the stored charge. To analyze the interaction of the two dominant discharging mechanisms, direct trap-to-band tunneling and thermally stimulated excitation, the influence of temperature on the charge decay in the temperature range from room temperature to 80K was investigated. The derived experimental results confirm the assumption, that both charge loss mechanisms should be considered in the extraction of the trap distribution. The typical approach is performing the measurements by elevated temperatures, when the thermal excitation becomes the dominant discharge mechanism [3]. In the present work the contrary approach was used: the retention measurements were carried out by low temperatures (80K) to evaluate the contribution of trap-to-band tunneling into the general charge loss. Retention measurements at low temperatures (80 – 300K) allowed probing the density distribution of "shallow" traps. Two methods of determination of the energy trap distribution from the retention measurements were then examined and the physically relevant was selected. Furthermore the dependence of the charge loss on the programming level and field conditions in tunnel oxide was investigated.

RuAl DEPOSITION BY MAGNETRON SPUTTERING

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Abstract :

The RuAl intermetallic compound is in many aspects, superior than most of the other B2 intermetallics, due to their favourable combination of physical and chemical properties, which make RuAl an ideal candidate for high temperature applications and as oxidation protective coatings. The influence of the thermal expansion coefficient of different materials is very important for the adherence of a coating. The RuAl thermal expansion coefficient (CTE) is nearly equal to that of Al₂O₃ in a large temperature range, which makes RuAl favourable for applications that require oxidation resistance. Its CTE is significantly lower than other intermetallics of interest, including FeAl, NiAl, CoAl and nickel-platinum aluminides. In this work Ru and Al were deposited over mirror polished 304 type stainless steel samples, through magnetron sputtering technique. Prior to deposition, the sample surface was cleaned using sputter etching, and the influence of this sputter etching time in the adhesion of the film was analyzed. The film-substrate behaviour was studied and the chemical composition was analyzed with EDX.

WEAR RESISTANCE OF ALUMINAS. DIELECTRICAL APPROACH

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Abstract :

During past year there has been considerable attention on wear of ceramics. It is now well accepted that most ceramics undergo a transition from mild to severe wear with a substantial increase in the wear rate. Several authors identified wear transitions depending on the environment. In mild-wear regimes plastic deformation and microcutting are dominant. Thermal-impact-induced brittle fracture is the predominant mechanism causing severe wear in the high -speed regimes. Considering the result above, the wear behaviour of ceramics seems to be very sensitive to the structure of the material (phase, porosity, toughness, hardness, grain size), and to the test parameters, such as environment and sliding speed. According to a new theory developed by Blaise and Le-Gressus based on the polaron approach of Mott, exoemission observed in ceramics subjected to electrical or mechanical strains can be interpreted as a process of mechanical relaxation of the polarization energy stored in a charged medium. Dielectrical breakdown and mechanical fracture have been observed simultaneously in dielectrics where trapped charges have been detected. Mechanical properties such as fracture, friction coefficient, wear of insulating materials are related to the mechanisms of storage and dissipation of energy. It is thus, sensible to study wear not only in mechanical terms but also in dielectric terms. In this work, a correlation is proposed between breakdown strength and mechanical properties (such as toughness and hardness). In order to evaluate a contribution of energy relaxation on friction and wear, we have studied the analogy between wear of our materials and their ability to store and dissipate polarization energy.

CRACK MODELING BY A HYBRIDIZATION WEIGHT FUNCTION APPROACH

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Abstract :

Elastic three dimensional mediums containing corner cracks and subjected to arbitrary distributed load are modeled using an extension of the hybridization weight function (HWF) approach. The HWF approach consists on subdividing the crack surface into two zones then use each of the chosen weight function reference solution in the zone where it is more efficient. This technique has previously been tested and proved its accuracy in static and fatigue problems. In the present work, we intend its extension for the computation of stress intensity factor (SIF) in mode I of corner cracks. For this purpose, and in order to take into account of the two free edge surfaces of the corner crack, the HWF is coupled to the point weight function method (PWFM). Validation tests are conducted on drilled plates with corner cracks at outer and inner borders of the plate's hole. The obtained results are in good concordance with the published results.

FORMULATION OF THE DNLR MODEL OF INELASTICITY IN LARGE DEFORMATIONS

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Abstract :

Recently, we proposed a thermodynamics-based modelling approach in order to describe the inelastic behaviour of a material. Based on the generalization of the Gibbs potential for situations out of equilibrium and on a spectral treatment of the material dissipation, this modelling doesn't use the concepts of yield surface, normality rule, and the internal variables. From the mathematical point of view, the constitutive equations correspond to a set of first order non linear differential equations whose numerical integration can be performed by using either implicit or explicit schemes. The implementation of the model in finite element softwares has also been achieved via the user subroutines. As an application, this modelling has been used, for example, to reproduce cyclic plasticity phenomena observed in the 316 stainless and in the Waspaloy metallic materials under proportional and non proportional complex loadings (Bauschinger, strain memory effects, additional hardening, softening),.Motivated by studying the problem of large deformations, which is of a great importance in several domains of engineering such as metal forming processes, we propose in this work an extension of the thermodynamically-based inelasticity model, developed and used under small perturbation hypotheses until now, for the large deformation framework.

OPTIMIZATION OF FABRICATION PARAMETERS OF ALUMINA/NICKEL ALLOY JOINTS FOR HIGH-TEMPERATURE APPLICATION

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Abstract :

Alumina was joining to the nickel alloy HAYNES ®214TM using a solid-state bonding technique with an intermediate nickel metallic foil. Experimentally, damages and cracks often are observed close to the metal/ceramics interface. Finite element analysis using an elastic-plastic-creep model has been extensively developed for predicting the thermomechanical behaviour of assembly. The goal of such modelling is to be able to design optimum interlayer and to use this design information to guide component fabrication. It is critical that the models be validated by comparison with experimental result. In this study, residual stresses distributions in the specimen were characterized experimentally using Xray diffraction (XRD) and indentation techniques. A good correlation between FEA analysis and experimental results is obtained. However, the Al₂O₃/Ni/HAYNESTM214® system always leads to high residual stresses. To solve this problem; work using a multilayer Cu/Ni/Cu joint associated with the Direct Copper Bonding method (DCB), by preoxidation of copper, allows reducing significantly the tensile residual stresses in ceramics and led to the fabrication of joints with reproducibly high strengths.

FRETTING WEAR DAMAGE OF HEXTOOLTM COMPOSITE DEPENDING ON THE DIFFERENT FIBER ORIENTATIONS

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Abstract :

Fretting has long been recognized as a source of wear and premature fatigue failures within mechanical parts. Fretting damage may occur whenever a junction between contacting parts is subjected to cyclic sliding micromotions, whose characteristic amplitudes are much less than the size of the contact. Such a contact loading can be induced either by vibrations or by the application of bulk fatigue stresses to one or both of the contacting parts [1].

Degradation of contact surfaces due to fretting wear is one of the principal industrial problems reducing service life of components. Wear induced by fretting is usually related to a three-stage phenomenon: (1) accommodation of the displacement in the upper layers of the two counterbodies; (2) detachment of particles from material; (3) third-body behaviour. The aim of this work is to study the viscoelastic behaviour of HexTOOLTM composite based on matrix polybismaleimide reinforced by carbon fibres and the influence of different fibre orientations in this composite on the evolution of fretting wear. On the one hand, the dynamic mechanical analysis (DMA) was conducted to characterize the viscoelastic behaviour of the composite subjected to a thermo mechanical loading as a function of temperature and frequency [2]. On the other hand, fretting tests were carried out using a geometry contact sphere /plane. These tests are based on a principle where a constant normal force (P) is applied while alternating displacement is imposed (δ). The latter is recorded along with the tangential force (Q) and the normal force. The evolution of the tangential force versus displacement allows plotting the fretting cycles called "log fretting". The information given on the log was used for the running condition fretting maps (RCFM). These maps showed various fretting regimes (partial slip, mixed and gross slip) and linked the stresses in the area of contact under the loading conditions. Moreover, an energy wear approach was developed. The influence of different fiber orientations of HexTOOL composite on the wear kinetics was shown.

**POLYURETHANEIMIDE MATERIALS CONTAINING TERTIARY AMINE GROUPS
FOR SO₂ GAS MICROSENSOR APPLICATIONS**

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Abstract :

Demand for SO₂ gas detectors is very important in the semiconductor industry and also in several applications related to environmental pollution. For these applications, surface acoustic wave (SAW) gas microsensors are very promising because they are cheap and very easy to implement. Nevertheless, the current challenge remains the enhancement of the microsensor selectivity. In this project, our main purpose is to develop new SO₂ gas microsensors with selective polymer top layers to obtain particularly high sensor characteristics. The literature references on the development of new polymer materials for SO₂ sensors are really scarce. Nevertheless, several publications on CO₂ (i.e. another acid gas) sensors have shown the great advantage of introducing Lewis basic sites in polymer materials for these applications [1]. On the basis of the expertise of LCPM on the synthesis of film-forming polyurethaneimide (PUI) materials containing Lewis basic sites [2,3], new PUI copolymers have been synthesized with a controlled number of tertiary amine groups with different basicities. This paper first describes their synthesis and characterization by several techniques including infra-red and advanced nuclear magnetic resonance spectrometry, viscometry, and size exclusion chromatography coupled to multi-angle laser light scattering for absolute molecular weight determination. The new PUI materials display excellent film-forming ability and polymer layers as thin as 150 nm have been easily obtained by spin-coating on silicon wafers. The first IJL results on the development of new SO₂ gas microsensors with selective PUI top layers are then presented. A first type of device based on a simple bilayer structure made of PUI on quartz was first tested but the corresponding surface acoustic wave was not suitable for the targeted application. A new SAW structure based on PUI/ZnO/Quartz was then successfully considered with ZnO playing the role of guiding layer.

EFFECT OF MICROSTRUCTURE ON THE SECONDARY ELECTRON EMISSION YIELD OF SAPPHIRES AND POLYCRISTALLINE ALUMINA CERAMICS

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Abstract :

Insulator materials are largely used in electrical and microelectronic applications. These materials are very sensitive to charged environment. When an insulator material is subject to electron irradiation, a fraction of incident and generated electrons are emitted from the surface while the rest remains at the sample. The ratio of the number of emitted electrons to incident ones represents the secondary electron emission yield. In this work, the evolution of the secondary electron emission yield during electron injection is used to characterise the effect of microstructure on the charging kinetic of sapphires and polycrystalline alumina ceramics. Experiments are performed using a Scanning Electron Microscope specially equipped with a secondary electron low-noise detector located under the objective lens just above the sample. The results show that the biased voltage applied to this detector influences the charging kinetics.

BEHAVIOUR OF GLASS CONTAINING ALKALI IONS UNDER E⁻ IRRADIATION

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Abstract :

This participation presents a study of electrical charging behaviour of a kind of glass containing alkali ions when submitted to electron beam irradiation in scanning electron microscope (SEM). Charge trapping and charge spreading in this glass have been examined using a time resolved current method. Our interest concerns more particularly the dynamic behaviour and the amount of the space charge build-up during and after electron irradiation under various primary beam energies. We examine also the effect of primary beam energy on the evolution of secondary electron emission yield (SEY) during irradiation and at saturation. As expected, the trapping and detrapping processes seem to be extremely dependent on the incident beam energy and on the chemical composition of the studied glass.

CHARGE REGULATION MECHANISM OF GROUNDED-COATED INSULATORS DURING THEIR IRRADIATION IN A SCANNING ELECTRON MICROSCOPE

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Abstract :

In this study, an original method is proposed for such a dynamical investigation of the trapping proprieties of a coated insulating samples submitted to electron irradiation in a scanning electron microscope. The secondary electron emission is restricted to the conductive coating, and this coating avoids complications associated with the change of the secondary electron emission which may blur the trapping measurements. This method, called "electrostatic influence method" is based on the measurement on the specimen holder situated on backside of the sample when electrons are trapped below the coating slice on the front side of the sample. This method allows to study charging and discharging phenomena and to determine accurately their time constants during electron irradiation.

Moreover, it is possible to estimate the trapped charge beneath the coating and the corresponding internal electric field in spite of the absence of external charging effects. This is to confirm that coating is not enough to stop ageing of a non conductive material (polymer, ceramic ,composite ...)

STRUCTURAL AND DIELECTRIC CHARACTERIZATIONS OF ZNO FILMS

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Abstract :

In the present work, we investigate the structural and the dielectric properties of ZnO films prepared from powders at temperature range 700°C-1000°C. For this purpose, we have used X-ray diffraction (XRD) and scanning electron microscopy (SEM) to study the effect of temperature on the morphology and the crystallinity of the obtained samples. XRD analysis shows a polycrystalline structure. The SEM observations show rough surfaces with ZnO grains having different sizes. Then, we have performed dielectric measurements by spectroscopic impedance at different temperatures and frequencies. The preliminary results show strong dependence of the permittivity constants and dielectric losses with the structural quality of the ZnO films.

MICROSTRUCTURE CHARACTERIZATION AND MECHANICAL PROPERTIES OF UFG AA6063

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Abstract :

Ultrafine grained (UFG) and nanocrystalline (NC) materials are of great interest in recent years. Severe plastic deformation (SPD) is an established method to produce UFG materials with extraordinary mechanical properties. In this work an equal channel angular pressing (ECAP) processed UFG aluminum alloy AA6063 was investigated. As the material properties are closely related to the microstructure, a careful characterization of the microstructure is necessary for the understanding of the material properties. Therefore, different electron microscopy methods were taken of use for the determination of the microstructure, in order to get more detailed information. Plastic deformation behaviour was investigated based on strain-controlled tensile and stand tensile as well as nanoindentation experiments. Strain rate sensitivity (SRS) is one important way to investigate the mechanical behaviour of UFG and NC face-cubic centered (fcc) metals at room temperature and to identify the deformation mechanisms, and this SRS behaviour was examined by strain controlled tensile and nanoindentation experiments. It was found that the

interaction between the dislocations and grain boundaries seems to be the main mechanism for the plastic deformation. Grain boundary is an important factor influencing the material properties and the plastic deformation process. Low angle grain boundaries (LAGBs) and high angle grain boundaries (HAGBs) have different abilities of the interaction between dislocations and grain boundaries which is an important factor influencing the plastic deformation process. Furthermore, the grain boundary sliding and grain boundary rotation were not found in the researched materials with a grain size in the UFG range.

BIPOLAR CHARGE TRANSPORT MODEL UNDER UNSTATIONARY TEMPERATURE GRADIENT IN POLYETHYLENE FILMS

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Abstract :

A bipolar charge transport model under unstationary temperature gradient is developed. The electronic charges are generated under applied DC voltage to polyethylene sample which is placed between two electrodes. The transport of charges is objected to trappingdetrapping and recombination mechanisms. The effects of the unstationary temperature gradient and the space charge packets generated by high DC voltage are shown on the space charge dynamics, the electric field distribution in the sample bulk and on the conduction current.

A CHART FOR WELDABILITY OF LOW ALLOY STEELS

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Abstract :

The weldability of a material is a poorly-understood concept because of the large number of variables involved in the welding process, as well as the context in which the concept is defined. However, the process variables may be divided into two groups - the processing parameters and the material properties – from which two characteristic indices may be defined. For the case of low alloy steels, a carbon equivalent is used to characterise hardenability during welding and the maximum hardness developed in the heat affected zone. The welding process itself is characterised in terms of its energy input. These two indices are used to construct a chart for weldability of low alloy steels. A physically-based analytical model is then used to establish a relationship between the indices, which is displayed on the chart as a contour for a predefined hardness, e.g. 350 HV. Empirical data are also plotted on the chart. Agreement between theory and data is found to be good. The chart may be used in an educational setting to explain the underlying principles of welding and weldability, and also in an industrial application to aid in an initial selection of materials and welding parameters, which may then be examined empirically.

CHARACTERIZATION OF POLY(ETHYLENE TEREPHTHALATE) USED IN COMMERCIAL BOTTLED WATER

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Abstract :

The aim of this study is to determine which compounds are present into drinking water packaged in poly(ethylene terephthalate) bottles and to know the origin of these substances in relationship with the material. A screening procedure was established for the detection of unknown compounds into bottled water. Liquid-liquid extraction (LLE) and solid-phase extraction (SPE) have been used to remove a large number of substances. Gas chromatography-mass spectrometry has been carried out to separate and to identify compounds in water extracts. A panel of 10 water bottles has been tested after exposure to extreme conditions of temperature and UV radiation to accelerate the possible migration of substances. At the same time, physico-chemical characterization of polymeric material has been performed namely RX scattering, calorimetric analysis, IRTF and low-frequency mechanical spectroscopy. The results thus obtained allow to understand in a better way the migration kinetics of molecules inside the polymer, it means the pollution of the bottled water.

MECHANICAL SPECTROSCOPY OF ROLLING OIL THIN FILMS ON COLD- ROLLED STEEL SHEET

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Abstract :

High resolution mechanical spectroscopy is a sensitive technique for detecting extremely fine traces of natural oils. Fine traces of groundnut oil can even be discovered in the form of fine debris left on the surface of cold-rolled steel sheets. It is shown that a characteristic mechanical loss spectrum occurs in the low temperature range (from 180K up to 280K) only in the two following cases, namely (i) if traces of the rolling oil are left on the surface of sheets and (ii) if the clean sheets are covered with a thin film of the rolling oil (or any other natural or mineral oil). It is clearly demonstrated that similar mechanical spectra induced by the presence of oil can be observed in the sub-resonant mechanical spectroscopy if the oil film is deposited on a very soft cellulose neutral substrate. Therefore the behaviour of the oil alone can be obtained with a high resolution. It will be demonstrated that it is possible to assign the mechanical loss phenomena to the steel sheet (like the Snoek-Koster effect above 500K) and oil separately. In particular, it is shown that the low temperature relaxation phenomena do not originate from a specific interaction of the steel surface with oil, as frequently reported in the literature.

**SYNTHESIS OF BULK MC6 GRAPHITE INTERCALATION COMPOUNDS
USING MOLTEN LITHIUM-METAL ALLOYS**

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Abstract :

Recently, the discovery of the superconducting properties of CaC₆ and YbC₆ compounds renewed the interest in binary MC₆ graphite intercalation compounds. It is particularly difficult to prepare the latter, by means of the reaction between pyrolytic graphite and metal vapour. Indeed, such a reaction is slow, often very incomplete (superficial intercalation) and moreover can lead to the formation of carbides due to too high reaction temperatures. If we except the case of lithium, the synthesis by direct plunging of pyrolytic graphite platelet into molten metal leads inevitably to the appearance of carbide, since the melting points of the considered metals are strongly higher than 700°C. We have developed a novel synthesis method, that consists in plunging a pyrographite platelet into molten lithium-M (M = Ca, Ba, Eu) alloys. By these means, it becomes easy to isolate the corresponding MC₆ binary intercalation compounds. This method needs reaction times, that do not exceed ten days. It allows also to obtain bulk samples, whose size can be quite large, so that the measurements of their physical properties become much easier and much more accurate. During such a synthesis, lithium plays two basic roles. In a first time, it leads to strongly reduce the reaction temperature, since, if the lithium concentration remains sufficiently high, the lithium alloys melt below 400°C, so that the formation of carbide is avoided. In a second time, it carries out the opening of the graphitic galleries, so that the intercalation of the second metal is largely made easier.

**TI-Nb ALLOY AS IMPLANT IN CONTACT WITH BLOOD:
INFLUENCE OF THE TOPOGRAPHY ON THE ENDOTHELIAL CELL RESPONSE**

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Atherosclerosis, an inflammatory disease, causes the growth of fibrous plaque which leads to the decrease of the artery luminal diameter, a phenomenon named stenosis. Stents, tubular implants in contact with the artery wall of endothelial cells, are widely used to restore the normal blood flow. However, this implantation causes damage to the endothelial cells leading to problems such as stent restenosis and thrombosis. These risks can be reduced if the damaged cells are fast and completely replaced with new endothelial cells (reendothelization).

Stainless steel such as 316L is currently one of the most used materials for stents. However, titanium and titanium alloys have an excellent corrosion resistance and are therefore good alternatives for stainless steel 316L that is susceptible to pitting corrosion.

The surface properties such as the chemical structure and also the topography are important parameters for the cell response.

In this work, the influence of different surface roughness on the endothelial cells is studied. We use TiNb10 as biomaterial because of its two-phase ($\alpha+\beta$) microstructure: Its surface can be structured by acid etching as the α -phase (Ti-rich) is preferentially etched compared to the β -phase (Nb-rich). The resulting roughness increases with the time of contact with the acid.

In our studies, we were able to show that the endothelial cell response improved when the roughness of TiNb10 decreased. The reendothelization may even be ameliorated by nanostructuring the surface.

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