

**FCT - Programa de financiamento plurianual**

Actualização de Unidades de I&D - 2004

Área reservada

[ Terminar sessão ]

ICEMS - INSTITUTO DE CIÊNCIA E ENGENHARIA DE MATERIAIS E SUPERFÍCIES (LISBOA) [84]

**Relatório Científico 2003**

[ Página Principal ]

Lacrado em 23-07-2004 10:05:00

**Indicadores de execução relativos a 2003 - Projecto Global**

<b>Indicador</b>	<b>Previsto</b>	<b>Realizado</b>
<b>Output indicator</b>	<b>Foreseen</b>	<b>Accomplished</b>
Livros	4	11
Artigos em revistas internacionais	78	86
Artigos em revistas nacionais	7	6
Em congressos científicos internacionais	74	100
Em congressos científicos nacionais	30	10
Relatórios	0	15
Organização de seminários e conferências	3	10
Teses de Doutoramento	5	2
Teses de Mestrado	5	2
Outra	0	0
Modelos	3	0
Aplicações computacionais	3	1
Instalações Piloto	0	0
Protótipos laboratoriais	3	0
Patentes	0	2

**Relatório científico por sub-projecto**

<b>Sub-projecto</b>	<b>Investigador responsável</b>
<b>Sub-project</b>	<b>Sub-project leader</b>
MATECHEMPRO	Maria Norberta Neves Correia Pinho
SMat	Luis Manuel Guerra Silva Rosa
PRECHAMAT	Rui Mario Correia Silva Vilar
<b>Total: 3</b>	

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**Relatório Científico 2003**

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**Sub-projecto**

**Sub-project**

Preparation and characterisation of multifunctional surfaces and thin films

**Investigador responsável**

**Project leader**

Rui Mario Correia Silva Vilar

**Resumo da actividade desenvolvida**

Preparation and characterisation of multifunctional surfaces and thin films

The objective of the project is to prepare and characterise multifunctional surfaces and thin films for various applications, aiming at establishing fundamental relations between structure, properties and processing parameters. These relations are compared with model predictions to uncover underlying mechanisms.

Tasks 1 and 2 - Surface and thin film preparation and characterisation

**Thin film preparation**

The materials investigated for functional thin film applications include metal oxides for spintronic applications, photonic materials for integrated optics, and wear and corrosion resistant coatings. Concerning magnetic oxides for spintronics, laser-assisted chemical vapour deposition (LCVD) and pulsed laser deposition (PLD) were used to grow thin films of chromium oxide (CrO<sub>2</sub>/Cr<sub>2</sub>O<sub>3</sub>) and magnetite on Si (100) and sapphire substrates. Also Co-doped TiO<sub>2</sub>-anatase films were deposited by PLD in order to obtain a high TC dilute magnetic semiconductor. The films were analysed by XRD, micro-Raman spectroscopy, SEM, AFM, Auger/XPS and RBS. Regarding Fe<sub>3</sub>O<sub>4</sub> films, conversion electron Mössbauer spectroscopy was also used. The resistivity and magnetic properties of the most promising samples were measured. The most promising results were obtained for magnetite thin films. In fact, it was shown that pure Fe<sub>3</sub>O<sub>4</sub> films can be deposited by PLD at temperatures as low as ~200 °C. This research work has been undertaken in close collaboration with ICSTM (London, UK).

In what concerns glasses and thin films for nanophotonics and integrated optics, silicate glass active optical planar waveguides doped with Er<sup>3+</sup> ions and Ag nanoparticles introduced by different methods were prepared by sol-gel processing. Characterization included visible m-line, Raman and photoluminescence spectroscopies, ellipsometry and RBS (ITN, Portugal). One of the main objectives was to determine the influence of the silver nanoparticles on the Er photoluminescence efficiency. The possible application of these waveguides as chemical optical sensors was examined. One and three-dimensional photonic bandgap structures were prepared by sol-gel processing for nanophotonics and integrated optics. Special effort was put into the fabrication of 1-D Fabry-Perot microcavities doped with Er<sup>3+</sup> and Yb<sup>3+</sup> ions and the characterization of the energy transfer processes when the active ions were confined in the cavity layers.

Within an industrial contract with TE&M abrasion resistant composites based on the Fe-Cr-C-NbC system were synthesised by laser cladding and tested for abrasion resistance. On the other hand, in a project in collaboration with PLASDAN, metastable Al-(Cr, Mo, Nb) alloys and Al-based MMC have been created by laser-alloying/particle injection, which were characterised in terms of microstructure and tribological behaviour. A variety of metastable aluminides were identified and their defect structure analysed by TEM. These materials are intended for application as coatings on Al-alloy rapid manufactured moulds, in order to enhance their lifetime. Cu nanocomposites, synthesised by mechanical alloying and laser cladding are being studied as coating materials, for sealing and thermal management applications

**Surface processing**

Surface functionalisation of metals such as aluminium alloys and coil coated galvanized steel is of prime technological interest. With this objective, inorganic-organic hybrid films based on silanes were prepared both by immersion and sol-gel processing. These procedures lead to the formation of nanostructured self-assembled monolayers of organofunctional silanes or to the deposition of nanoparticles on surfaces that improve the corrosion resistance of the pre-treated substrates and paint adhesion. On line with this, special attention to corrosion resistance of the painted materials was given. Characterisation was performed by techniques such as SVET, Scanning Kelvin Probe, Electrochemical Impedance spectroscopy, SEM, TEM, AES/XPS and AFM. The results obtained help to understand fundamental aspects related with surface functionalisation, performance and durability of the modified surfaces in aggressive environments. Fundamental studies on the passivity of metals and alloys were also carried out. The evaluation of the semiconducting properties of the films, in conjunction with their chemical properties led to the development of important models that strongly contribute to understand protection mechanisms.

Laser-assisted surface microtexturing is being investigated as a tool to control prosthesis-body interactions and drug release from biomaterials such as hydroxyapatite, HA-bioglass and Ti-alloys (in collaboration with INEB). Different types of surface morphology are observed depending on the processing parameters. In certain conditions, a columnar topography develops with a high specific surface area and enhanced drug

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release capability. The use of excimer lasers to drill narrow tunnels in human enamel and dentin to access internal carious lesions and the influence of laser processing on the decay of biological dental materials has been investigated too.

### Task 3 Modelling

In this Task a physico-computational model and computer codes that relate the transient thermal field in a part build up by laser freeform manufacturing and solidification parameters and microstructure with the processing parameters is being developed and applied as a tool for LFFM process optimisation. The model allows the solidification path and solid-state diffusive and displacive phase transformations during the consecutive heating-cooling cycles associated to track overlapping and their impact on the final properties to be predicted and residual stresses, dimensional changes and distortion to be calculated.

Three-dimensional photonic bandgap structures were first modelled by transfer matrix techniques, which allowed the simulation of the width and centre wavelength of the stop bands and the optical cavity modes of the projected photonic bandgap structures.

### Indicadores de execução 2003

	Previsto	Realizado
<b>A - Publicações</b>		
Livros	1	3
Artigos em revistas internacionais	40	45
Artigos em revistas nacionais	2	4
<b>B - Comunicações</b>		
Em congressos científicos internacionais	26	42
Em congressos científicos nacionais	5	3
<b>C - Relatórios</b>	0	3
<b>D - Organização de seminários e conferências</b>	1	9
<b>E - Formação Avançada</b>		
Teses de Doutoramento	2	0
Teses de Mestrado	2	2
Outra		
<b>F - Modelos</b>		
<b>G - Aplicações computacionais</b>	0	1
<b>H - Instalações Piloto</b>		
<b>I - Protótipos laboratoriais</b>		
<b>J - Patentes</b>		

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**FCT - Programa de financiamento plurianual**

Actualização de Unidades de I&D - 2004

Área reservada

[ Terminar sessão ]

**ICEMS - INSTITUTO DE CIÊNCIA E ENGENHARIA DE MATERIAIS E SUPERFÍCIES (LISBOA) [84]**

**Relatório Científico 2003**

[ Página Principal ]

[ Relatório Científico 2003 ]

**Sub-projecto**

**Sub-project**

Structural Materials: processing, analysis and modelling

**Investigador responsável**

**Project leader**

Luis Manuel Guerra Silva Rosa

**Resumo da actividade desenvolvida**

2003 Report

Sub-Project: Structural Materials: processing, analysis and modelling

**Task 1: Cellular Materials**

During 2003, the activities developed within Task 1 focussed on the following areas:

**1) Liquid foams**

- Problems related to the energy of 2D foam clusters.
- Instabilities in 2D and 3D bubble clusters.
- Shapes of liquid films/bridges.
- Tensile testing of 3D foams using the Wilhelmy balance to obtain force-displacement curves.
- Effect of Plateau borders (triple film junctions) on the equilibrium and rheology of 2D and 3D foams.
- Foam drainage.

**2) Cellular materials**

- Structural and mechanical characterization of novel insulating hybrid aerogel foams.
- Cork quality evaluation.
- Biaxial tests of cellular materials, including cork.
- Permeability of cork and plastic stoppers to gases.

**Task 2: Multiphase Polymeric Systems and Biopolymers**

1) The Nuclear Magnetic Resonance Laboratory and its affected team were involved in the following activities:

The study of dental polymeric materials. Relevant mechanical and physicochemical properties of novel composite resins were studied, as a function of the monomer composition and filler loading. Conventional (three-steps), self-priming and self-etching dental adhesives were evaluated using liquid- and solid-state NMR techniques and stray-field MRI, in the presence of the solvent component, in order to obtain the volumetric contraction and to characterize the more mobile constituents in the photo-polymerized materials.

The study of glass-ionomers used in Dentistry. Borosilicate glasses were synthesised and analysed by  $^{11}\text{B}$ ,  $^{27}\text{Al}$  and  $^{29}\text{Si}$  solid state NMR spectroscopy in order to study the influence of  $\text{CaO}$  and  $\text{Al}_2\text{O}_3$  incorporations in the glass structure (B and Al speciation, relative concentrations of 4-/3-coordinated boron and 4-/6-coordinated aluminium). Solid state NMR studies and compressive strength tests were performed on the glass ionomer cements prepared with the glass samples. A set of Na-Sr-phosphate glass and glass-ceramic samples, with general formula  $x\text{SrO}:(0.55-x)\text{Na}_2\text{O}:0.45\text{P}_2\text{O}_5$ , were prepared and analysed by solid state  $^{31}\text{P}$  NMR and X-ray powder diffraction in order to obtain structural information on the  $\text{Sr}^{2+}$  incorporation.

The molecular mobility of decanol monolayers on a NaA zeolite external surface was studied by solid-state  $^1\text{H}$  NMR spectra.

Chromium/zeolite catalysts were studied by  $^{29}\text{Si}$  NMR MAS spectroscopy and the mobility in the MHz range was obtained as a function of Cr oxidation state.

The development of theoretical models capable of predicting the photo-polymerisation behaviour of the materials under investigation (e.g., depth of cure, degree of conversion and shrinkage), as a function of suspending phase (monomer solution) chemical composition, reinforcement (particulate) distribution and volume contents, and radiation exposure dose.

2) Most of the activity of the Laboratory of Rheology was dedicated to the rheology of multiphase systems, in particular:

Rheology of liquid foams: constitutive equations and its experimental validation in strong flows.

Rheology of asphalt bitumen mixtures: a new class of asphalt modifiers based on reactive polymers was

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developed. The new mixtures were tested in different conditions: transient, steady and dynamic tests. Both Laboratories were also involved in the characterization of polyolefins synthesized via metallocene catalysis, in cooperation with colleagues of the ICEMS Catalysis group.

**Task 3: Ceramic materials and composites**

The research work in 2003 has covered the following areas:

- 1) Ceramic matrix composites and carbon-carbon composites - validation of test procedures and assessment of factors that affect the measurements, namely of test methods for characterising reinforcements (multifilament tows consisting of small-diameter SiC, Al<sub>2</sub>O<sub>3</sub> and carbon fibres) and test methods to determine the fiber/matrix interfacial frictional shear stress (using minicomposites); evaluation of damage in composites through cyclic tensile tests; development of statistical models for the distribution of strength data.
- 2) Ornamental stones - characterisation of stones (microstructure and mechanical properties, evaluation of dynamic test methodologies for assessing the elastic constants of granitic rock materials); characterisation of diamond tools (wear behaviour and mechanical properties); and study of new stone processing technologies using diamond tools, including the optimisation of polishing processes and investigating the use of new inspection techniques for assessing stone surface quality and study of new diamond tools for polishing stones.
- 3) Materials processing by solar furnace - improvement of the IST solar furnace, that included the integration of a remote control in a Labview environment; evaluation of tests using different heating and cooling cycles inside the reaction chamber; comparison between the sintering of consolidated (dense) ceramic bodies using solar radiation, and those produced in conventional furnaces; and continuation of the investigation concerning the synthesis reactions under solar radiation, and characterization of the reaction products.

**Task 4: Fatigue of materials and composite materials**

- 1) Dynamic multiaxial loading of metallic materials, where new experiments on low cycle fatigue tests under tension and torsion loading on three metallic materials were completed. Simulation of the elasto-plastic behaviour of these materials was studied using finite element techniques.
- 2) Fatigue crack growth of carbon steels under variable amplitude loading was continued with a special emphasis on the study of crack closure influence on fatigue crack growth. A new testing methodology was introduced, concerning the study of fatigue crack growth and crack closure under vacuum conditions, in order to eliminate the oxide induced crack closure.
- 3) The study of the behaviour of the repair of composite materials with patches when subjected to repeated compression loading was concluded.
- 4) A new subject related with composite materials was initiated concerning the study of mixed mode fracture analysis of laminated composite materials. A new testing apparatus was designed and preliminary tests concerning mode I / mode II fracture were achieved.
- 5) A new subject related with the bending behaviour of reinforced sandwich beams (with foam core) was initiated, where both testing and finite element analysis are carried out. Different reinforcements were tested and different non-linear theories were applied in order to model the bending behaviour of the reinforced sandwich beams.

**Task 5: Fatigue and Fracture of Engineering Materials and Structures**

Research projects were in progress in 2003:

- 1) "Fatigue creep of Udinet 720 LI", RTO/NATO, P 123.
- 2) "Surface integrity for fatigue life extension", RTO/NATO, P 131.
- 3) "Improving the fatigue performance of welded stainless steels", EU ECSC Programme, 7210 - PR301/F3.
- 4) "Fatigue design of exhaust conducts of naval gas turbines", Portuguese Navy, ICEMS/UME.

**Indicadores de execução 2003**

	Previsto	Realizado
<b>A - Publicações</b>		
Livros	2	6
Artigos em revistas internacionais	18	20
Artigos em revistas nacionais	2	1
<b>B - Comunicações</b>		
Em congressos científicos internacionais	25	38
Em congressos científicos nacionais	20	7
<b>C - Relatórios</b>	0	10
<b>D - Organização de seminários e conferências</b>	1	1
<b>E - Formação Avançada</b>		
Teses de Doutoramento	2	1
Teses de Mestrado	2	0
Outra		
<b>F - Modelos</b>		
<b>G - Aplicações computacionais</b>		
<b>H - Instalações Piloto</b>		
<b>I - Protótipos laboratoriais</b>		
<b>J - Patentes</b>	0	1

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## FCT - Programa de financiamento plurianual

Actualização de Unidades de I&D - 2004

Área reservada

[ Terminar sessão ]

ICEMS - INSTITUTO DE CIÊNCIA E ENGENHARIA DE MATERIAIS E SUPERFÍCIES (LISBOA) [84]

### Relatório Científico 2003

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#### Sub-projecto

#### Sub-project

FUNCTIONAL MATERIALS IN ADVANCED CHEMICAL PROCESSES

#### Investigador responsável

#### Project leader

Maria Norberta Neves Correia Pinho

#### Resumo da actividade desenvolvida

Summary of the 2003 activity report

The synthesis and characterization of functional materials - catalysts, membranes and electrodes - and their applications in advanced Chemical / Electrochemical Processes have been carried out, according to the tri-annual planning 2003 - 2005 of the project, in three main directions:

#### 1. CATALYTIC REACTION PROCESSES

##### 1.1 - Catalytic Functionalization of Light Alkanes

The oxidative dehydrogenation of *i*-butane to isobutene, a valuable chemical intermediate with important industrial applications, was studied over silica supported nickel molybdates by XRD, temperature programmed reduction, TEM, SEM, temperature programmed desorption and activity tests.

##### 1.2 - Selective Oxidation of Olefins over Mixed Oxides

Correlation and interpretation of experimental data on the synergy effects between  $\delta$  and  $g$  phases of bismuth molybdates in the selective oxidation of 1-butene.

##### 1.3 - Polymerisation and Technology of Polymerisation of Olefins over Metallocenic Catalysts

Studies on the transition metal catalyzed copolymerization of vinyl based monomers. Development of novel applications for waste products craft paper industry derivatives: 3,7-dimethylocta-1,6-diene and 5,7-dimethylocta-1,6-diene. Development of new supported metallocene systems to be used in ethylene/diene copolymerisation. Development of novel functional polymeric materials. Correlation studies of composition and properties.

##### 1.4 - Hydrotreatment of Petroleum Distillates over Cobalt and Nickel Molybdates

The deep and ultra-deep hydrodesulfurization of thiophene, benzothiophene and 4,6-dimethyldibenzothiophene was studied over industrial cobalt - nickel molybdate catalysts in a joint project with the portuguese petroleum company Petrogal.

##### 1.5 - Selective Catalytic Oxidations of Methanol and Ethanol

Preparation of super acid Zr-W catalysts using novel techniques. Systematic physical-chemical characterization of the prepared catalysts. Testing of such catalysts with the oxidative dehydrogenation of ethanol. Test of the same catalysts as supports for the deposition of MoO<sub>3</sub> that exhibited in such circumstances very interesting performances for the oxidation of ethanol to acetaldehyde. Preparation and characterization of Zr-Ce and Ti-Ce mixed oxide catalysts.

#### 2. Membranes and Membrane Separations and Processes(MSP)

##### 2.1 Preparation, functionalization and characterization of polymeric membranes

Bi-soft segment urethane/urea membranes were synthesized with soft segments of Polypropylene/Polybutadiene (PU/PBDO) and Polypropylene/ Polydimethylsiloxane (PU/PDMS).The PU/PBDO membranes were extensively characterised through Infrared Spectroscopy in Attenuated Total Reflectance mode (ATR-FTIR) , Positron Annihilation Spectroscopy(PALS)(collaboration with ICEMS Coimbra) and Scanning (SEM) and Transmission Electron Microscopy (TEM).The ATR-FTIR and PALS results led to two publications - *Macromolecules*, 36, 4195 (2003); *Radiation Physics and Chemistry*,68,573(2003). Hard/soft/soft segment phase separation was correlated to gas permeation properties.

The membranes were tested for gas permeation and for Bio and Hemo-compatibility for their application in extra-corporal blood oxygenators .

Surface charge properties and zeta-potential of commercial membranes were investigated. Preparation of cellulose acetate membranes with control of pore sizes was carried out and their permeation performance investigated.

### 2.2. Modelling of Flow and Mass Transfer at Membrane/Fluid Interfaces

Nanofiltration(NF) and Ultrafiltration(UF) have been the subject of Computer Fluid Dynamics(CFD) simulations and permeation experiments. These were performed in a flat-sheet cell. For NF the cell feed channel accommodates ladder-type spacers typical of spiral-wound modules. The results were reported in a publication-Desalination, 157, 395(2003)-in three communications to international meetings (Malta, Spain) and in a MSc. Thesis. The UF results are relative to the effect of high permeation rates on the feed flow structures and mass transfer in protein (lysozyme)/salt(NaCl)/water systems. They are reported in two publications(one submitted and another in preparation) and in a communication to an international Congress.

### 2.3. Development of Membrane Hybrid Processes

Integration of UF/NF in drinking water production (four communications to international meetings (Germany, Spain, Italy) and two articles submitted for publication).

Wine clarification and tartaric stabilization were investigated leading to a PhD Thesis: "Optimization of Membrane - Based Processes for Wine Clarification and Tartaric Stabilization. Influence of the Macromolecular/Ionic Composition", Fernando Gonçalves, Instituto Superior Técnico, Universidade Técnica de Lisboa, November 12, 2003( Prémio CUF - menção honrosa); three publications(Journal of Food Engineering, 59, 229(2003); Carbohydrate Polymers 53, 297(2003); Carbohydrate Polymers, 53, 1(2003), two communications to international congresses (Spain, France ).

Production of wines with low alcohol content led to a patent.

UF/NF for Cheese Whey Recovery led to the submission of a publication and to a communication in an international Congress (Spain) and another in a national meeting.

Protein Recovery from Fish Meal Wastewaters by Ultrafiltration

Desalination of Brackish Water by Reverse Osmosis

Downstream Processing of Fermentation Broths for the Optimization of the Isolation of Antibiotics

### 3. Electrochemistry of Materials

During the period of 2003 we pursued studies of PEMFC and AFC systems. We were particularly concerned with the development of mischmetal-based hydrogen storage electrode alloys (HSA). The electrochemical properties of PEMFC MEA using differently prepared HAS MI Ni<sub>3</sub>.65CO<sub>0.85</sub>Al<sub>0.3</sub>Mn<sub>0.3</sub> alloys as anode electrocatalysts instead of Pt/C alloys were studied, and the materials were developed. Different surface treatments were investigated and the electrochemical and electrocatalytic properties of the HAS electrodes were characterised.

#### Indicadores de execução 2003

	Previsto	Realizado
<b>A - Publicações</b>		
Livros	1	2
Artigos em revistas internacionais	20	21
Artigos em revistas nacionais	3	1
<b>B - Comunicações</b>		
Em congressos científicos internacionais	23	20
Em congressos científicos nacionais	5	0
<b>C - Relatórios</b>	0	2
<b>D - Organização de seminários e conferências</b>		
<b>E - Formação Avançada</b>		
Teses de Doutoramento	1	1
Teses de Mestrado	1	0
Outra	0	0
<b>F - Modelos</b>	3	0
<b>G - Aplicações computacionais</b>	3	0
<b>H - Instalações Piloto</b>	0	0
<b>I - Protótipos laboratoriais</b>	3	0
<b>J - Patentes</b>	0	1

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