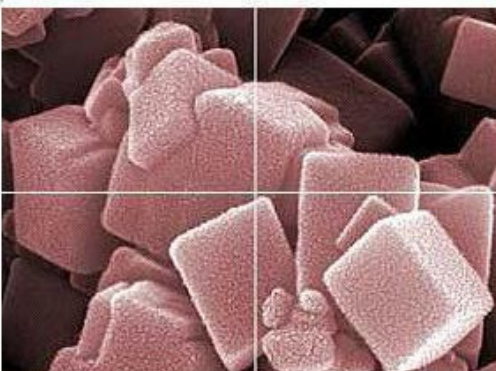


ACTIVITY REPORT 2004

AND

ACTIVITY PLAN 2005



**ASSOCIATE LABORATORY
CENTRE FOR RESEARCH IN CERAMICS AND
COMPOSITE MATERIALS**

UNIVERSITY OF AVEIRO



ACTIVITY REPORT 2004
AND
ACTIVITY PLAN 2005

ASSOCIATE LABORATORY

CENTRE FOR RESEARCH IN CERAMICS AND COMPOSITE MATERIALS



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SPONSORS



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INTERREG IIB
'Materials network for the Atlantic Area' -
MNAA



University of Aveiro



Portuguese Republic Government

FCT **Fundação para a Ciência e a Tecnologia**
MINISTÉRIO DA CIÊNCIA, TECNOLOGIA E ENSINO SUPERIOR



**Programa Operacional 'Ciência, Tecnologia e
Inovação'**

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SECTION 1

FACTS AND NUMBERS

The **Centre for Research in Ceramics and Composite Materials (CICECO)**, established in the University of Aveiro in the beginning of 2002, is one of the Portuguese Associate Laboratories.

The performance of **CICECO** in the past three years has been extremely positive: Research Staff, Scientific Output and Research Projects, all have steadily increased in number.

An overview of the research team over the past three years is given in Table 1:

Table 1

	2002	2003	2004
Professors and Lecturers	47	47	50
Full Time Researchers	5	9	12
Post-Doctoral Associates	23	22	29
Collaborators	16	13	12
PhD Students	54	60	61
MSc Students and Other Students	26	44	54
Laboratory Technicians	4	8	5
Administrative Personel	1	3	5
TOTAL	176	206	228

On 31ST December 2004 **CICECO** hosted 228 people (full list at the end of Section 1), a 10.7% increase over 2003 and 29.5% over 2002. In particular, the number of Full-Time Researchers, Graduate Students and Post-Docs rose significantly.

The 2004 CICECO's Scientific Production comprises 11 PhD and 7 MSc Theses terminated and 288 papers published in SCI Journals (Table 2).

The number of 2004 papers published in SCI Journals increased 26.3% over 2003 and 40.5% over 2002; The average contribution per Professor or Full-Time Researcher is now 4.7. The number of SCI papers published in large (≥ 5) Impact Factor Journals has also increased to 4.

Section 4 gives a complete account of **CICECO's** Scientific Production in 2004.

Table 2

		2002	2003	Corrigendum 2003	2004
Theses	MSc	10	8	-	7
	PhD	14	13	-	11
Books	Editions	0	0	-	1
	Chapters	4	14	-	12
SCI Papers	IF ≥ 5	1	3	-	4
	IF < 5	204	221	4	284
TOTAL		233	259	4	319
Average SCI Paper per Professor and Researcher		3.94	4.07	-	4.65

Over the past three years 13 Patents have been filled.

The number of projects in progress in 2004 was 103, similar to 2003 (100) but a major increase over 2002 (62). The main source of funding continues to be FCT (National Science Foundation) and the European Funding Agencies and Programmes (E. Commission, FEDER, INTERREG IIIB).

The following European Programmes have started: FAME – Advanced Materials Engineering of Hybrids and Ceramics (NOE); Innovation and Sustainable Development in the Fibre Based Packaging Value Chain (IP); Materials Network for the Atlantic Area.

Section 5 lists all the Projects funded.

IMPORTANT EVENTS

On September 22nd **CICECO** was evaluated by a panel of foreign experts appointed by FCT. In general, this mid-term assessment confirmed that CICECO is giving the right steps in order to fulfil its vision of becoming a leading European Research Centre in Materials Science and Technology.

On October 22nd and 23rd **CICECO** organised a national science and technology meeting aimed at establishing networks of scientists, with the participation of about 400 Portuguese scientists of all fields. This meeting was reputed a success.

In collaboration with a group from the Chemistry Department, **CICECO – CDTM** decided to create its first spin-out company (FoodMetric) which will target the food industry companies.

CICECO – CDTM also started a protocol with the Biotech Company Alfama (Lisbon) whereby it will sell intellectual property rights concerning certain potentially Bio-Active Molecules.

An Erasmus Mundus Master Programme has been started with the Universities of Aalborg and Hamburg: EMMS, Joint European Master Programme in Materials Science.

On 7-8 January 2005 we organised the 2nd Meeting of CICECO Researchers (3 invited talks, 15 oral presentations, 60 posters).



CDTM Centro de Design
e Tecnologia de Materiais

CENTRE FOR MATERIALS DESIGN & TECHNOLOGY (CDTM)

In 2004 CDTM carried out a range of activities.

Conferences & Workshops

The main objective of these events was to advertise CDTM in regional and national companies and discuss specific issues of interest to them.

- S1** - Building mortars: new challenges and solutions (University of Aveiro, 17-03-2004)
- 04** - Rheology of Mortars (University of Aveiro, 9-09-2004)

R&D and training needs of companies

CDTM visited several companies in order to identify joint industry/academic research programmes meeting regional needs:

- Pavicentro – Pré-fabricação, S.A. (12-11-2004)
- Topcer – Ind. de Cerâmica, Lda (16-11-2004)
- Bemorporce – Fábrica de Porcelanas, S.A. (16-11-2004)
- Amedida, Lda (19-11-2004)
- Esmalticer – Esmaltes Cerâmicos, Lda (17-11-2004)
- Teka Portuguesa – Equipamentos de Cozinha, Lda (23-11-2004)
- Vulcano – Termo-domésticos, S. A. (2-12-2004)
- Porcel, S.A. (16-12-2004)

Applied R&D activities

Technical assistance was provided to the companies:

- Cerutil – Cerâmicas Utilitárias S. A.
- Caima – Indústria de Celulose S. A.
- Indasa S. A.
- Durit, Metalurgia Portuguesa do Tungsténio S.A.
- Weber-St Gobain SA
- Recer, SA
- Modicer, S.A.
- Repsol, S.A.
- Petrobras – Petróleo Brasileiro S.A.

Development of training programmes for industrialists

Preparation of the training programme: ‘Environment and sustainable development’ in collaboration with a local trade association (‘Associação Industrial de Águeda’)

R&D Projects

The following projects, aimed at developing close-to-the-market products, have been financed:

- ‘Fotossensibilizadores imobilizados como novos materiais no tratamento de águas’, FCT POCTI/CTM/58183/2004
- ‘Projecto da casa do futuro’ - Sub-projecto de águas interiores (Aveirodomus – Projecto ADRI – Acções para o desenvolvimento Regional de Base Industrial)

Materials Network for the Atlantic Area - MNAA

CDTM started MNAA, a European network financed by INTERREG IIIB. In the frame of this programme, 5 student grants have been awarded to carry out applied research, in collaboration with companies.

‘Wax-fibre based phase change materials’, *Pavicentro & Somague*;

‘Study of building rehabilitation mortars’, *Weber-Cimenfix*;

‘New sub-micrometric hardmetal (WC-Co) grades with enhanced erosive wear resistance’, *Durit*;

‘Substitution of talc by calcium hydroxide in tile ceramic formulations for fast firing’ *Recer*;

‘Preparation of powders, membranes & films of microporous luminescent silicates’.

CDTM Staff Training Courses

- New Product Development (COTEC - 22 to 26-03-2004)
- Turning Technologies into Businesses – COHiTEC (COTEC – 30-03-2004 to 8-06-2004)
- Creating Business out of Science (ASTP – 14 to 15-10-2004)

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SECTION 2

ACTIVITY REPORT 2004

Research carried out at **CICECO** encompasses three distinct areas and nine lines of study:

Area 1 - ADVANCED MICRO- AND NANO-STRUCTURED MATERIALS FOR COMMUNICATIONS TECHNOLOGIES

Inorganic Multifunctional Materials and Organic-Inorganic Hybrids
Electroceramics
Magnetostructural Modulation of Strongly Correlated Electric Materials
Advanced Molecular and Supramolecular Materials

Area 2 - ADVANCED MATERIALS FOR INDUSTRIAL APPLICATIONS

Reactive Ceramic Components for Process Control
Ceramic Composites and Ultra-Hard Coatings for Mechanical Applications

Area 3 - CHEMISTRY AND TECHNOLOGY OF POLYMERIC AND LIGNOCELLULOSIC MATERIALS AND BIOMATERIALS

Macromolecular Materials and Lignocelullosics
Biomedical and Biomimetic Materials
Process Development and Optimisation

AREA 1 - Advanced Micro- and Nano-Structured Materials for Communications Technologies

INORGANIC MULTIFUNCTIONAL MATERIALS AND ORGANIC-INORGANIC HYBRIDS

New Microporous Materials. Much work concentrated on the synthesis and characterisation (structure and photoluminescence, PL) of novel microporous lanthanide silicates, for example, AV-9 materials ($\text{Na}_4\text{K}_2\text{LnSi}_{16}\text{O}_{38}\cdot 10\text{H}_2\text{O}$, $\text{Ln}=\text{Tb}^{3+}, \text{Er}^{3+}$). Tb-AV-9 is an efficient X-ray scintillator (using $\text{CuK}\alpha$ radiation). In addition dehydrated Er-AV-9 is an interesting C-band infrared emitter. Microporous zirconium silicate umbite is also being evaluated as a suitable host matrix for hosting Eu(III) ions (in the pores). Upon calcinations, this material yields synthetic (dense) wadeite which seems to be also an interesting PL material. The synthesis, structural characterisation (single-crystal XRD data) and PL properties of novel microporous $[\text{Na}_3(\text{EuSi}_6\text{O}_{15})\cdot 2(\text{H}_2\text{O})]_n$, (AV-21) have been reported. The structure of this material is related with that of mineral sazhinite. A considerable amount of research dealt with the preparation of films and membranes of microporous materials (luminescent or not). A paper has been published on membranes and films of titanosilicate ETS-10 supported on alumina and stainless steel. Attempts to engineer magnetic centres into microporous materials resulted in the publication of the first example of a stoichiometric large-pore framework Cr(III) silicate catalysts. The synthesis of a new synthetic microporous zirconosilicate with the structure of mineral tumchaite has been reported.

Due to the intrinsic importance of microporous aluminosilicates and aluminophosphates (zeolites) as molecular sieves, ion exchangers, catalysts and catalyst supports, we have successfully applied recent advances in mathematical tiling theory to the systematic enumeration of 4-connected crystalline networks (i.e., networks in which each atom is connected to exactly four neighbours), which resulted in over 900 topologies distributed among uni-, bi- and trinodal frameworks. For each of these structures treated as silica polymorphs, relevant optimised structural parameters (unit cell dimensions and crystallographic symmetry), framework energies relative to α -quartz and volumes accessible to sorption have been calculated. As only a fraction of the mathematically generated networks are expected to be chemically feasible, we devise of an effective “filtering” process, based on the calculated physical properties of each framework, which can identify the most plausible frameworks.

Mesoporous Materials. The cyclopentadienyl complex $(\text{RC}_5\text{H}_4)\text{Mo}(\text{CO})_3\text{Cl}$ [$\text{R} = -\text{C}(\text{O})\text{N}(\text{H})-(\text{CH}_2)_3\text{Si}(\text{OEt})_3$] was prepared and immobilized in the mesoporous silica MCM-41 by covalent grafting. A supported dioxo complex of the type $(\eta^5\text{-C}_5\text{H}_4\text{R})\text{MoO}_2\text{Cl}$ was subsequently prepared by oxidative decarbonylation of the tethered tricarbonyl complex using *tert*-butylhydroperoxide (TBHP). The oxidized material is an active catalyst for the liquid phase epoxidation of cyclooctene with TBHP as the oxygen source. Similar catalytic results were obtained using the tethered tricarbonyl complex directly as a pre-catalyst since fast oxidative decarbonylation occurs under the reaction conditions used. For both systems, the desired epoxide was the only product and the initial activities were about $13 \text{ mol mol}_{\text{Mo}}^{-1} \text{ h}^{-1}$. The solid catalysts were recycled several times. Some activity was lost from the first to second runs, but thereafter tended to stabilize. In another study, the acetonitrile

complex $[\text{Mn}(\text{NCCH}_3)_6][\text{B}(\text{C}_6\text{F}_5)_4]_2$ was immobilized in MCM-41 functionalized with a pyrazolylpyridine ligand. A metal loading of 0.72 wt-% (0.13 mmol g^{-1}) was achieved.

Layered Materials. The hydrothermal synthesis and structural characterization of layered lanthanide silicates, $\text{K}_3[\text{M}_{1-a}\text{Ln}_a\text{Si}_3\text{O}_8(\text{OH})_2]$ (M) Y^{3+} , Tb^{3+} ; Ln) Eu^{3+} , Er^{3+} , Tb^{3+} , and Gd^{3+}), named AV-22 materials, have been reported. Er-AV-22 is a room-temperature infrared phosphor, while Tb- and Eu-AV-22 are visible emitters with output efficiencies comparable to standards used in commercial lamps. The structure of these materials allows the inclusion of a second (or even a third) type of Ln^{3+} ion in the framework and, therefore, the fine tuning of their photoluminescent properties. For the mixed $\text{Tb}^{3+}/\text{Eu}^{3+}$ materials, evidence has been found of the inclusion of Eu^{3+} ions in the interlayer space by replacing K^+ ions, further allowing the activation of Tb^{3+} -to- Eu^{3+} energy transfer mechanisms. The occurrence probability of such mechanisms ranges from 0.62 ($a=0.05$) to 1.20 ms^{-1} ($a=0.1$) with a high energy transfer efficiency (0.73 and 0.84, respectively).

Two papers have been published on layered double hydroxides (LDHs) intercalated with non-steroidal anti-inflammatory drugs and with indomethacin. In the latter case, a pharmacological study was also reported.

Zn-Al LDHs intercalated by ferrocenecarboxylate (FcCOO) and 1,1'-ferrocenedicarboxylate [$\text{Fc}(\text{COO})_2$] anions were prepared by co-precipitation from aqueous solution. Powder X-ray diffraction (XRD) indicated that the material $\text{Zn}_2\text{Al-Fc}(\text{COO})_2$ contained a monolayer of guest anions with the longest dimension of the anions perpendicular to the host layers, resulting in a basal spacing of 15.5 \AA . The material $\text{Zn}_2\text{Al-FcCOO}$ exhibited a basal spacing of 20.0 \AA , consistent with the formation of a bilayer of organometallic guest species. Dehydration of $\text{Zn}_2\text{Al-Fc}(\text{COO})_2$ prompts reorientation of the ferrocene guest anions, resulting in the formation of a collapsed phase with an interlayer separation of 12.3 \AA . The structural transformation is fully reversible on rehydration. In a second study, Zn-Al LDHs intercalated by terephthalate (TPH), biphenyl-4,4'-dicarboxylate (BPH) and 2,2'-bipyridine-5,5'-dicarboxylate (BPY) anions were synthesized either by direct co-precipitation from aqueous solution (TPH, BPH) or by ion-exchange of a precursor material in nitrate form (BPY). The basal spacing for the Zn,Al-TPH intercalate was 14.6 \AA , indicating that the guest anions stack to form a monolayer with the aromatic rings perpendicular to the host layers. For the LDH intercalates containing BPH and BPY anions, the observed basal spacing of about 18 \AA suggests that the anions are tilted slightly with respect to the host layers. The material Zn-Al-BPY was examined as a “solid ligand” for the immobilization of the dioxomolybdenum(VI) complex $\text{MoO}_2\text{Cl}_2(\text{THF})_2$. Molybdenum K-edge EXAFS analysis could not substantiate the formation of a supported complex of the type $\text{MoO}_2\text{Cl}_2(\text{N-N})$, but instead indicated the formation of unidentate-bridged entities of the type $[\text{O}_2\text{Mo}-\text{O}-\text{MoO}_2]$ with a metal-metal separation of 3.29 \AA . The molybdenum-containing LDH is an active and recyclable catalyst for the selective epoxidation of olefins, and the stability is superior to that reported for a material prepared by immobilization of $\text{MoO}_2\text{Cl}_2(\text{THF})_2$ in the mesoporous silica MCM-41 derivatized with bipyridyl groups.

Nanostructured Materials. Synthetic well-defined SiO_2 particles were used as substrates to grow ZnS nanoparticles at the silica surfaces. These composite particles were investigated as precursors to produce ZnO supported on sub-micron silica, by a simple calcination process. The structural and optical properties of the supported ZnO phase were investigated. The ZnO emission is dominated by the typical green band at 2.5 eV commonly associated with intrinsic defects. Cadmium sulfide and cadmium selenide/polymer nanocomposites were prepared via *in situ* radical polymerization in miniemulsion. Organically capped CdE (E=S, Se) quantum dots (QD's) were used as the starting materials and ensembles of these dots were encapsulated with no need of further surface treatment. The use of two polymer matrices was investigated: poly(styrene) (PS) and poly(*n*-butyl acrylate) (PBA). In both cases, homogenous nanocomposites were obtained and their optical properties were investigated by visible absorption and photoluminescence spectroscopy. Quantum size effects were assigned to the nanocomposites indicating the integrity of the individual QD's upon polymer encapsulation using the miniemulsion process. Rod-like particles of the analogues of mineral natrophilite (NaMnPO_4) have been prepared by hydrothermal synthesis. Micro-patterning of such crystals has been performed by a judicious choice of the reacting conditions, yielding open micro-tubules with internal diameters ranging from 10 to 30 μm . A plausible mechanism for the tubular microstructure formation has been proposed. The use of NaMnPO_4 micro-tubules as host materials for metallic (*eg.* gold) particles has been investigated. Single crystals of $\text{K}_2\text{V}_3\text{O}_8$ have been synthesized using a mild hydrothermal method. The synthetic method allows morphological micro-patterning of well aligned tubular-windows on sheet-like $\text{K}_2\text{V}_3\text{O}_8$ single crystals. The lateral dimensions of the tubular-windows range from 2 to 0.1 μm and are *ca.* 80 μm in depth.

Novel Pigments. Cellulosic fibres were used to obtain well-defined BiVO_4 pigment particles using a simple controlled precipitation method; microscopy analysis suggests that the BiVO_4 particles grow from nucleation sites located in the cell wall structure and inside the lumen of fibres.

Polyoxometalates. The work on new compounds with polyoxometalates and organic dipolar molecules was continued. New compounds were prepared and characterised with (a) the anions $[\text{XM}_{12}\text{O}_{40}]^{n-}$, X = P (n = 3), Si (n = 4), M = Mo, W, and S-arginine and urea, and (b) the Fe-substituted polyoxotungstates $[\text{XW}_{11}\text{Fe}(\text{H}_2\text{O})]^{n-}$, X = P (n = 4), Si (n = 5), with aminoacid derivatives. Compounds with the parent Keggin anions were prepared for further studies on their optical properties. The new compounds with Fe were used in catalytic studies of oxidation of hydrocarbons and terpenes. The tetrabutylammonium salts of the Keggin-type polyoxotungstates $[\text{XW}_{12}\text{O}_{40}]^{n-}$, $[\text{XW}_{11}\text{O}_{39}]^{n-}$, $[\text{XW}_{11}\text{V}^{\text{V}}\text{O}_{40}]^{n-}$ and $[\text{XW}_{11}\text{M}^{\text{III}}(\text{H}_2\text{O})\text{O}_{39}]^{n-}$, X = P, Si, B and M = Fe or Mn, proved to be effective catalysts for oxidation of different substrates with hydrogen peroxide, in acetonitrile. Catalytic studies went on during the year referred in this report. In an article on catalytic studies with the Keggin anions with X = B it was shown that an adsorbed single-layer of the hybrid tetrabutylammonium salts of the anions $[\text{PW}_{11}\text{O}_{39}]^{7-}$, $[\text{PW}_{11}\text{M}(\text{H}_2\text{O})]^{4-}$, M = Fe, Mn and $[\text{PW}_{11}\text{Co}(\text{H}_2\text{O})]^{5-}$, can be fabricated on the surface of a glassy carbon electrode by the

droplet evaporation methodology. These chemically modified electrodes were stable and their preparation was reproducible and easy to perform. The electrochemical features of the immobilized polyanions were studied.

Several lanthanide (Ln(III): Eu, Sm and Tb) complexes having a polyoxotungstocobaltate as ligand were prepared. This inorganic ligand contains Co(II) in a tetrahedral environment. The presence of Ln(III) and Co(II) in the polyoxotungstates may confer an important combination of properties, namely luminescence and magnetism. The coordination of Ln and Co in the same oxo cluster was obtained for the first time. Hybrid silica materials incorporating the polyoxotungstates with Ln(III) and Co(II) were also prepared. The first Ce^{IV} complex with two distinct Keggin-type lacunary polyoxotungstophosphates was synthesised, $\text{H}_7\text{K}_{10}[\text{Ce}_2(\text{PW}_{10}\text{O}_{38})(\text{PW}_{11}\text{O}_{39})_2] \cdot 28\text{H}_2\text{O}$, belonging to a new class of lanthanide-substituted polyoxometalates in which the bilacunary $[\text{PW}_{10}\text{O}_{38}]^{11-}$ anions show an unprecedented bridging structural motif. The structure of the compound was elucidated by single-crystal X-ray diffraction and consists of discrete V-shaped anionic complexes, $[\text{Ce}_2(\text{PW}_{10}\text{O}_{38})(\text{PW}_{11}\text{O}_{39})_2]^{17-}$, *ca.* 1.5x2.2 nm in size and constructed from bilacunary $[\text{PW}_{10}\text{O}_{38}]^{11-}$ and monolacunary $[\text{PW}_{11}\text{O}_{39}]^{7-}$ polyanions bridged by Ce^{IV} cations. A novel supramolecular compound of europium(III), $\{[\text{Eu}(\text{CH}_3\text{OH})_6(\text{H}_2\text{O})_2][\text{PMo}_{12}\text{O}_{40}]\} \cdot (\text{C}_{14}\text{H}_{20}\text{O}_5)_2 \cdot (\text{CH}_3\text{OH})_2 \cdot (\text{CH}_3\text{CN})_2$ (**1**) (where $\text{C}_{14}\text{H}_{20}\text{O}_5$ is benzo-15-crown-5), was synthesised and structurally characterized by single-crystal X-ray diffraction. It is the first supramolecular compound containing a lanthanide (Eu^{3+}) metal centre together with the polyoxoanion ($[\text{PMo}_{12}\text{O}_{40}]^{3-}$) and crown ether ($\text{C}_{14}\text{H}_{20}\text{O}_5$) moieties. Interestingly, in the compound Eu^{3+} is coordinated to eight solvent molecules (six of methanol and two of water), with the resulting complex being strongly hydrogen-bonded to two moieties of benzo-15-crown-5 and crystallisation solvent. A novel supramolecular organic inorganic adduct containing α -Keggin-type $\text{PW}_{12}\text{O}_{40}^{3-}$ anions and benzo-15-crown-5 molecules was also prepared.

Novel Luminescent Systems. Research on novel lanthanide (Ln) luminescent systems based on Ln complexes and its incorporation in nanomaterials was continued. Research concerned the coordination chemistry of Ln with derivatised [60]fullerene ligands and with aromatic ambidentate ligands, exploring the possibility of formation of multidimensional Ln compounds. The incorporation of those Ln compounds into nanosized SiO_2 and other substrates was explored considering the possible applications of the resultant materials in optical devices. The luminescence and structural properties were studied. A series of new Ln compounds containing a benzo-15-crown-5 derivatised [60]fullerene was prepared and, based on the crystallographic and photoluminescence evidence, the related $[\text{Tb}(\text{H}_2\text{O})_3(\text{NO}_3)_2(\text{acac})] \cdot \text{C}_{14}\text{H}_{20}\text{O}_5$ [where acac^- = acetylacetonate and $\text{C}_{14}\text{H}_{20}\text{O}_5$ = benzo-15-crown-5] supramolecular adduct was further used as a model for a possible coordination sphere of the lanthanide cations.

Novel luminescent materials were prepared by introducing a new Eu^{3+} complex of 2,6-dihydroxybenzoic acid (2,6-Hdhb) into a silica gel made by the sol-gel method. The crystal structure of the resulting complex $[\text{Bu}_4\text{N}]_2[\text{Eu}(\text{2,6-dhb})_5(\text{H}_2\text{O})_2]$ was determined. Photoluminescence measurements were performed for the isolated Eu(III) 2,6-dihydroxybenzoate complex and also for the related silica composite material.

Adducts of the type $\text{Ln}(\text{NTA})_3 \cdot \text{L}$ [$\text{Ln} = \text{Eu}, \text{Gd}$; $\text{NTA} = 1\text{-(2-naphthoyl)-3,3,3-trifluoroacetone}$; $\text{L} = 1,4\text{-diaz-1,3-butadiene}$ $\text{RN}=\text{CHCH}=\text{NR}$ ($\text{R} = p\text{-tolyl}, o\text{-tolyl}$)] were prepared. *Ab initio* calculations were carried out in order to predict coordination geometries, and also to interpret the vibrational spectra. The room temperature (RT) PL spectra of the two Eu complexes are composed of the typical Eu^{3+} red emission, assigned to transitions between the first excited state ($^5\text{D}_0$) and the ground multiplet ($^7\text{F}_{0-4}$). Based on the RT emission spectra and lifetime measurements, the quantum efficiencies of the $^5\text{D}_0$ Eu^{3+} excited state were estimated and found to be quite low (e.g. 2-3%). The low efficiencies were attributed to the presence of thermally activated non-radiative channels involving ligand-to-metal charge-transfer (LMCT) states. Indeed, a low-lying LMCT band was detected for the Eu complex containing the ligand *p*-tolyl-DAB.

Crystal Engineering of Organic-Inorganic Hybrids. A 3D diamondoid framework, $[\text{Cd}(\text{NDC})(\text{H}_2\text{O})]$ (where $\text{NDC}^{2-} = 2,6\text{-naphtalenedicarboxylate}$), was isolated using hydrothermal syntheses, which were properly optimised to obtain a highly crystalline material: (1) the temperature programme for the reactive period has been systematically changed and an optimal programme selected; (2) as the reactive mixture is a typical ternary system (composed of triethylamine, Cd^{2+} and H_2NDC), the composition was varied in order to isolate the conditions under which a pure homogeneous phase is obtained. Under this context a novel layered structure was also discovered for high concentrations of TEA, and formulated as $[\text{Cd}_2(\text{NDC})(\text{OH})_2]$. Such synthetic approach was successfully applied to other more complex systems containing highly flexible organic ligands, namely 1,2-bis(4-pyridyl)ethane (BPE) and 4,4'-trimethylenedipyridine (TMD), which, along with trimesic acid (H_3BTC), allowed us to demonstrate that the increased flexibility of TMD (when compared to that of BPE) leads, on the one hand, to a larger variety of possible structures (i.e., *supramolecular isomerism*) and, on the other, to significantly less crystalline materials. Four compounds were described: $[\text{Cd}_{1.5}(\text{BTC})(\text{BPE})(\text{H}_2\text{O})_2] \cdot (\text{H}_2\text{O})$, a 3D two-fold interpenetrated net exhibiting an unusual $(9,^3_4)$ topology; $[\text{Cd}(\text{HBTC})(\text{TMD})_2] \cdot 8.5\text{H}_2\text{O}$, a 2D framework with (4,4) topology; $[\text{Cd}(\text{HBTC})(\text{TMD})(\text{H}_2\text{O})] \cdot 4.5\text{H}_2\text{O}$, a 3D two-fold interpenetrated diamondoid framework; $[\text{Cd}_2(\text{BTC})(\text{TMD})_2(\text{NO}_3)] \cdot 3\text{H}_2\text{O}$, a compact 8-connected 3D modular structure with a highly unusual $3^6 4^{22}$ topology. Another main achievement was the use of centrosymmetric $[\text{V}_2\text{O}_2(\text{pmida})_2]^{4+}$ units [where $(\text{pmida})^4$ is N-(phosphonomethyl)iminodiacetate] as robust building blocks for the construction of novel frameworks. First, the use of Co^{2+} cations led to the isolation of neutral $[\text{Co}_2\text{V}_2\text{O}_2(\text{pmida})_2(\text{H}_2\text{O})_{10}]$ species, which were then used as secondary building units by selectively replacing the coordinated water molecules by bridging organic 4,4'-bipyridine ligands (4,4'-bpy). Two isostructural, porous and non-interpenetrated 3D frameworks with topology identical to that of the NbO net were isolated: $[\text{MVO}(\text{pmida})(4,4'\text{-bpy})(\text{H}_2\text{O})_2] \cdot (4,4'\text{-bpy})_{0.5}$ (where $\text{M} = \text{Cd}^{2+}$ or Co^{2+}). Another milestone was the preparation and structural characterisation (particularly, ^1H , ^{13}C , ^{31}P and ^{51}V solid-state NMR studies) of a novel phosphovanadate layered structure intercalated by 4,4'-bipyridinium cations, $(\text{C}_{10}\text{H}_{10}\text{N}_2)[(\text{VO}_2)_4(\text{PO}_4)_2]$. This material is formed by an unprecedented secondary tetrametallic V^{V} building unit exhibiting a distorted cubane-type structural motif, which is the first of its kind assembled by only VO_6 octahedra.

Organic-Inorganic Hybrids Lacking Activating Centers. The discussion about the recombination mechanisms and the chemical nature of the emitting centers subjacent to the white-light emission of sol-gel derived amine-functionalized hybrids lacking metal activator ions, such as those based on 3-aminopropyltriethoxysilane (APTES), 3-glycidyloxypropyltrimethoxysilane (GPTES) and on urea and urethane precursors, continued during the last year. The white-light photoluminescence (PL) results from a convolution of the emission originated in the NH (NH₂) groups of the urea or urethane bridges (APTES- and GPTES-based hybrids) with electron-hole recombinations occurring in the siloxane nanoclusters. These two components reveal a radiative recombination mechanism typical of donor-acceptor pairs, mediated by some localized centers. Photoinduced proton-transfer between defects such as NH₃⁺ and NH⁻ (GPTES- and APTES-based hybrids) or NH₂⁺ and N⁻ (di-ureasils and di-urethanesils) is proposed as the mechanism responsible for the NH-related component. Electron paramagnetic resonance data suggest that the specific PL mechanism subjacent to the component associated with the siliceous nanodomains involves oxygen-related defects.

The effect of the synthesis method (conventional sol-gel and carboxylic acid derived solvolysis) on the luminescent features, in particular, on the emission quantum yield (QY) of di-urea and di-urethane cross-linked hybrids was investigated. Di-ureasils with three different polymer chains were prepared using acetic acid. Furthermore, the influence of the carboxylic acid was also investigated for the shorter polymer chain di-ureasil. Structural characterization indicated a similar structure for all the hybrids, independent of the synthesis process and selected carboxylic acid. All materials are efficient room temperature (RT) white-light emitters with emission quantum yields (QY) 6-20%. The emission QY of the hybrids prepared through carboxylic acid solvolysis are 27-35 % higher than those calculated for the di-ureasils and di-urethanesils synthesized via the conventional sol-gel technique. This was attributed to the presence of a larger number of non-bonded NH urea- and urethane-groups in the hybrids prepared by carboxylic acid solvolysis, illustrating the key-role played by the synthetic method on the extent and magnitude of hydrogen bonding involving urea and urethane linkages. The use of different carboxylic acid doesn't lead to significant changes either in the structural and PL features.

New Hybrid Materials. The interest of chitosan-based hybrids lies in their chemical versatility, which allows tailoring novel functionalities accomplished by cross-linkages between the different polymer groups and the inorganic component. The chitosan/siloxane hybrids were synthesized by a sol-gel derived carboxylic acid solvolysis. Structural characterization confirmed that the derivatives are bifunctional hybrids, in which urea and urethane bridges covalently bond chitosan to the polysiloxane network. From the bioactivity tests, it can be concluded that the apatite formation mainly depends on the amount and distribution of the silanol groups in materials structure. The PL results of the hybrids based on low-molecular weight chitosan furnish unequivocally evidences for the presence of a new band centred about 340 nm (with higher energy and long lifetime) relatively to the characteristic emission of pure low-molecular weight chitosan). Since this component is associated with electron-hole recombinations mediated by donor-acceptor pairs arising from silicon-related defects at the surface of the siliceous nanodomains, the features observed in the high-energy region of the PL spectrum of the chitosan-based hybrids,

could be connected with the condensation degree of the inorganic domains. The siloxane-chitosan hybrids exhibit therefore interesting photoluminescent characteristics that may be adequate to their use as optical probes for applications *in vivo*.

The magnetic characterization of bio-inspired iron-doped di-ureasils continued during 2004. Magnetic studies in natural ferritin and iron oxide (ferrihydrite and others) nanoparticles in organic-inorganic hybrids were performed. The effect of the particle size and the magnetic moment distributions on the magnetic properties and the influence of hydrochloric acid (HCl) addition on the structure, thermal and magnetic properties were investigated. The structure, magnetic and thermomechanical properties of the hybrids were affected in acid catalysis. The magnetic results show that in both samples (with and without HCl) antiferromagnetic nanoparticles coexist with small clusters/isolated ions. In the sample without HCl addition, larger particles dominate the magnetic behaviour while the opposite occurs for the sample prepared using HCl catalyst.

In order to investigate the ability of different hybrid materials to efficiently incorporate lanthanide ions, protecting them from non-radiative channels, thus enhancing the respective PL features the following organic-inorganic hybrids were synthesised and investigated: i) Aminosils doped with $\text{Eu}(\text{CF}_3\text{SO}_3)_3$ - Eu(III)-based aminosil hybrids whose structure is formed by a siliceous network containing pendant amine terminated propyl chains were prepared. The effective interaction between the Eu^{3+} ions and the host matrix depends markedly on the amount of Eu^{3+} incorporated and was modelled in terms of a local-field perturbation representing the ion's nearest ligands interaction potential. ii) Mono-urethanesils and di-urethanesils doped with $\text{Eu}(\text{CF}_3\text{SO}_3)_3$ - FTIR and Raman spectroscopy and two-dimensional correlation spectroscopic analysis were employed to examine the anionic local environment in *mono-urethanesils* doped with europium triflate. The results obtained provide conclusive evidence that, in terms of ionic association, the level of complexity of these xerogels is very high. In all the compounds the triflate ions exist “free”, weakly coordinated and forming cross-link separated ion pairs. At higher guest salt concentration in addition to all these species contact ion pairs occur. The hybrids are efficient RT white-light emitters due to the convolution of a large broad-band in the blue-green spectral region with the straight lines typical of the Eu^{3+} ions. In order to investigate the Eu^{3+} local-environments the PL features and lifetimes were measured at 14K. The results allowed us to establish the presence of three distinct cation local sites, $\text{Eu}^{3+}/\text{O}=\text{C}$ (urethane cross-link), $\text{Eu}^{3+}/\text{O}-\text{C}-\text{C}$ (polyether chains), and weakly coordinated $\text{Eu}^{3+}/\text{CF}_3\text{SO}_3^-$ ionic pairs, a result that is in perfect agreement with the FT-IR and FT-Raman conclusions.

C60 Phase Transitions Under High-Pressure. The studies of polymeric phase transitions of C60 under high-pressure and high-temperature using X-ray diffraction techniques at the European Synchrotron Radiation Facility were continued. Most time has been spent treating raw diffraction data and subsequent structural determination of the novel 3D polymeric structures. The last task is rather difficult since several phases as well as twinning contribute to the diffraction patterns. The complexity of these patterns renders the structural determination not straightforward.

Development of Spectroscopic Techniques. Work has been continued on the development of novel techniques for the study of half-integer quadrupole nuclei in solids. A review paper on progress of multiple-quantum MAS NMR spectroscopy has been published in a book by Springer-Verlag.

The technique of SERS (Surface-Enhanced Raman Scattering) is an effective surface analytical technique which can allow the detection of submonolayer adsorbate coverage. The potential of SERS in the study of the interaction of metal nanocrystals with molecular adsorbates has been explored, in particular for the investigation of adsorption modes and orientation of molecules on the surfaces, with relevance in heterogeneous catalysis and nanoparticle assembly studies.

ELECTROCERAMICS

Microwave Ceramic Dielectrics. Regarding $\text{La}(\text{Mg}_{1/2}\text{Ti}_{1/2})\text{O}_3$ (LMT) based microwave dielectric ceramics, a PhD thesis on the subject was terminated.

Particular studies were carried on solid solutions of LMT with CaTiO_3 , SrTiO_3 and BaTiO_3 , previously processed by a chemical route as nano-powders, and obtained as dense, single-phase ceramics in all the range ($0 \leq x \leq 1$). Far infrared spectroscopy of all these compositions was also performed. Extrapolations from the far infrared region down to the microwave range allowed the study of contributions controlling the microwave properties. Processing, structure and dielectric properties of these systems were reported.

Promising microwave dielectric properties were obtained for $x=0.5$ in $(1-x)\text{LMT}-(x)\text{BT}$ ceramics ($\epsilon_r=60$; $\tau_f=-2$ ppm/K; $Q_f=9600$). The $x=0.5$ compositions for all solid solutions of LMT with CT, ST and BT were the most close-to-zero τ_f compositions. In these systems, permittivity and τ_f follow a hyperbolic-type dependence with composition. LMT-BT and LMT-ST systems were investigated regarding the structure-properties relations. Structure transformations were followed by Rietveld refinement of XRD data. For $x \leq 0.1$, XRD showed evidence for 1:1 ordering between Mg and Ti cation sub-lattice. For $x \leq 0.3$, compounds presented both in-phase and anti-phase tilting. However, for $0.3 < x < 0.7$, only anti-phase tilting of the oxygen octahedra was present. In the LMT-ST system similar changes were observed. In these systems, microwave dielectric loss was correlated with the one calculated from far infrared data and it was possible to trace its origin with lattice anharmonicity phenomena and also to particular lattice defects. Microwave losses, assessed also by far infrared spectra, are essentially intrinsic and for the case of LMT-BT an additional mechanism based on ionic conduction is present at rich BT compositions. Sintering in air and oxygen atmospheres was also performed during the quest to enlighten these structure-properties relations.

A singular relaxor behaviour was also detected for $x > 0.9$ LMT-BT ceramics, which is interesting if one thinks that it is a non-lead material. Studies on ferroelectric-relaxor transitions in this system were initiated. Radio frequency dielectric response measurements were done for those compositions and results were reported. Air and oxygen sintered ceramics were characterized at several frequencies in the 12-550 K temperature range. The dielectric permittivity maximum shifts by 300K towards temperatures lower than the Curie point for pure BT ferroelectrics ($\sim 400\text{K}$). Furthermore, the onset of relaxor behaviour was noticed with a strong frequency dependence of both real and imaginary parts of the dielectric complex function.

Besides the macroscopic level, dielectric response was also studied in this system at the nanoscale level with the use of piezoresponse force microscopy (PFM). Results compare the bulk ceramics response with that of individual grains. In addition to permittivity and P-E hysteresis loop measurements as a function of temperature, local electromechanical properties were measured (PFM). It was found that temperature evolution of the dielectric properties detected by macroscopic methods is similar to that observed at the nanoscale level (grain by grain).

Ferroelectric Ceramics. The objective of the work on the microstructural design of PZT bulk ceramics is to study the microstructural design of PZT bulk ceramics to obtain dense materials with large mean grain size, superior to 20 μm . The effect of the addition of a PbO-SiO₂ glass on the microstructural evolution during the sintering of PZT ceramics was investigated. The effect of glass amount, composition and thermal cycle was studied. Dielectric characterisation of the samples was also done.

Single phase SrBi₂Ta₂O₉ (SBT) ceramics were fabricated by template grain growth (TGG), using plate-like anisometric SBT single-crystals with a dominant (001)-orientation of a major face. Template particles, of size 50x50x5 μm^3 were previously obtained by the self-flux solution method, and then imbedded in a fine matrix with a 3w% of Bi₂O₃ excess using a uniaxial pressing technique. Highly dense SBT ceramics (>95%) were obtained for different sintering conditions (temperature 1150-1250 °C, sintering time 2-24 h). Bimodal microstructure was obtained for template ceramics, with predominantly large elongated grains (100x10 μm) perpendicularly aligned to the pressing direction. A preferential orientation of the platelets with the c-axis along the pressing direction was observed. The study of the dielectric properties revealed the influence of the texture degree on the electric properties of the obtained SBT ceramics. Permittivity values of the templated ceramics measured along the pressing direction are lower than those of the unseeded ceramic, but exceed them if measured in a perpendicular direction. This fact was attributed to the increased contribution of the highly polarizable a,b-plane allowed by the favourable alignment of grains in the direction of the applied electric field.

A new procedure for preparing SBN powders, starting from SrCO₃, Bi₂O₃ and Nb₂O₅ was developed. Pure SBN is obtained at 820 °C while using the solid state route a temperature in excess of 1000 °C is required. The individual particles obtained by this new method have submicrometric size but the existence of agglomerates is still an issue of concern for the purpose of using these powders as seeds. Improvements of the method are underway aiming at overcoming this problem.

The domain structures of ceramics with the morphotropic phase boundary composition 0.68PFW–0.32PT were analyzed in-situ by TEM as a function of temperature from 16 to 300 K. A core shell structure was detected at room temperature, in which the core of the grains was Ti- and the shell W- rich with respect to the bulk composition. At 16 K, a macrodomain state is observed throughout the volume of the grains, but at >250K a domain-free shell is formed around a central core that still exhibits strong domain wall strain contrast in two beam conditions. The presence of the core-shell microstructure and the growth of the core on-cooling was correlated with the appearance of (h00)/(00l) peak splitting in XRD traces as temperature decreases.

Ferroelectric Fibers, Single Crystals and Films. PZT fibers were prepared by sol-gel. Acrylic and methacrylic acids were used to acidify the PZT precursors. The effect of the type and content of acid on the macroscopic, structure, crystallization and microstructure of sol-gel precursors and PZT fibers was analysed, compared and discussed. Because long polymeric chains are formed in the precursors acidified with acrylic or methacrylic acids longer gel and ceramic PZT fibers were pulled, and the pure perovskite phase was obtained after heat treatment at a considerably lower temperature, 550 °C. The nature of the polymerisation, the type and structure of the polymeric chain generated in the precursor and the amount of organics to be released when organic acids are used influence the shape and crack formation process of the fibers. Due to the linear shape of the polymeric chains formed in the sol acidified with acrylic acid, long, round and cracked free PZT fibers were prepared from the acrylic acid precursor. While long, rectangular and cracked PZT fibers were prepared from the methacrylic acid precursor, in which a branched furcate type structure was identified. The obtained results indicate that the length and strength of PZT fibers can be improved by acidifying the PZT precursors with organic acids, namely with acrylic acid.

The ferroelectric and dielectric anisotropy in high-quality $\text{SrBi}_2\text{Ta}_2\text{O}_9$ single crystals produced by a high temperature self-flux solution method was investigated. The crystals were naturally oriented with [001] direction perpendicular to the major face and edges parallel to [110] axes. The dielectric and ferroelectric properties of the crystals were investigated along the c-axis and in the ab-plane of the orthorhombic unit cell. The ferro-paraelectric phase transition was observed at $T_C = 355$ °C. The maximum permittivity measured in the ab-plane (~ 1500) was an order of magnitude greater than that along the c-axis (~ 135). The Curie constant $\approx 4.4 \times 10^4$ °C was derived from the dielectric temperature curve. Saturated hysteresis loop was obtained along the [110] direction thus allowing estimation of spontaneous polarization $\approx 20 \mu\text{C}/\text{cm}^2$ along polar axis in this material. The study of the domain structure configuration of the produced SBT single crystals was carried out by optical observations with polarized light. Complex domain patterns with mutually orthogonal domains in (a,b)-plane were observed. The twin boundaries with rounded borders may correspond to microtwinning at the scale of the size of elementary cell. The domain structure consisting of fine antiparallel domains oriented along [100] direction was also investigated by Piezoresponse Force Microscopy (PFM). Fine 180° domains are directed in both (a,b) and (b,c) planes having periodicity of about 350 nm.

PZT thick films in the thickness range of 5 to 200 μm on 20 μm copper and 25 μm platinum foils were prepared by electrophoretic deposition (EPD) for application as embedded passive components. The EPD process was conducted in glacial acetic acid medium and the effects of deposition parameters, such as dc voltage values, processing times and suspension aging on the film thickness and composition stoichiometry were evaluated. It was shown that aging of the suspensions increases the current flow continuously. However, the effect of aging on the variation of the thickness of the films is not continuous. A thickness maximum was observed for the suspension aged for 7 days. The observed variations were related with the chemical modifications occurred in the suspension (solution and particles), that take place as the metal oxides leach out of the powder particles. Using fresh suspensions, with controlled particle size and optimized voltages and deposition times, reproducible PZT thin/thick films with desired thickness and improved properties were prepared. Monophasic, homogeneous and dense films with 8 μm of thickness were obtained at 1150 °C / 30 min, with a dielectric permittivity of ~ 1330 , loss tangent of 0.05,

remanent polarization of $24\mu\text{C}/\text{cm}^2$ and maximum polarization value of $38\mu\text{C}/\text{cm}^2$, respectively. These dielectric data are comparable with those of bulk PZT ceramics, strongly suggesting that PZT thick films prepared by EPD on metal foil are potential candidates for embedded components (capacitors and sensors) in various electronic components.

$\text{Ba}_x\text{Sr}_{1-x}\text{TiO}_3$ (BST, $x=0.5$) thin films were prepared on $\text{Pt}/\text{TiO}_2/\text{SiO}_2/\text{Si}$ substrates using a diol based sol-gel method. Two different solvents were employed, ethylene glycol and propylene glycol (1,2 propanediol). The influence of the solvent on the sol structure, microstructure development and dielectric properties of BST thin films was analysed and discussed. It was observed that films prepared with ethylene glycol show higher values of the dielectric permittivity ($\sim 40\%$ higher) and lower values of dielectric losses, when compared with films prepared from propylene glycol. Dielectric permittivity is 459 and 302 at 1 kHz for films prepared with ethylene glycol and 1,2 propanediol, and annealed at 800°C , respectively. IR studies revealed that the stabilization of titanium precursor with propylene glycol is incomplete. During the phase formation process a higher content of a second phase is formed for the propylene glycol delivered films. However, microstructure analyses revealed smaller grain size and surface roughness and higher density for films prepared with ethylene glycol. Moreover a (100) preferential orientation was observed for these films. These differences were attributed to the relative high boiling point, latent heat of vaporization, and to the linear molecular structure of ethylene glycol. Ethylene glycol as a solvent allows the preparation of BST thin films with improved dielectric properties.

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Raman investigation of ferroelectrics and related materials, X-ray diffraction (XRD) and Raman scattering in sol-gel derived $\text{Pb}_{1-x}\text{Ca}_x\text{TiO}_3$ (PCT) thin films are studied as a function of Ca content x . It is found that Ca^{2+} addition to PbTiO_3 films results in a gradual decrease of tetragonality of the perovskite lattice accompanied with a considerable modification of the Raman spectra. A significant difference in Raman spectra of PCT films and available reference data for single crystals ($x=0$) is explained in terms of the large internal stress, small grain size, and structural disorder produced by Ca^{2+} substitution. It is found that the shift of the frequency of ferroelectric soft mode correlates well with the variation of tetragonality for $x \leq x_c=0.4$. The concentration dependence of the shift is strongly non-linear being qualitatively different from that observed earlier in other A-site doped lead titanate-based systems. According to XRD data, the averaged crystal structure for $x_c \approx 0.4$ appears as pseudo-cubic, while Raman scattering revealed short-range order tetragonal distortion. The structural characteristics of PCT films are linked to their electromechanical properties measured earlier by interferometric technique.

PZT films of the composition close to the morphotropic phase boundary were deposited onto standard $\text{Si}/\text{SiO}_2/\text{Ti}/\text{Pt}$ substrates using a modified sol-gel process. The preparation conditions were optimized in order to obtain high-quality films at sufficiently low temperature ($T_a=500^\circ\text{C}$). The dielectric, ferroelectric and piezoelectric properties of the films were then measured as a function of the annealing temperature and the number of distillations to evaluate their suitability for micromechanical applications. The maximum values of the longitudinal charge and voltage piezoelectric coefficients were $d_{33} \sim 65 \text{ pm/V}$ and $g_{33} \sim 4 \cdot 10^{-3} \text{ Vm/N}$, respectively. The results indicate that the piezoelectric properties are improved and became saturated with increasing number of distillations and are almost independent on T_a . Only moderate decrease of the

piezoelectric response with frequency suggests that the investigated PZT films can be used in high-frequency piezoelectric applications.

Incipient ferroelectrics. The systems, $\text{Sr}_{1-x}\text{Mn}_x\text{TiO}_3$ ($0 \leq x \leq 0.15$ and $x = 1$) and $\text{SrTi}_{1-y}\text{Mn}_y\text{O}_3$ ($0 \leq y \leq 0.1$), were synthesized by conventional mixed oxide method. The grain size differs markedly for $\text{Sr}_{1-x}\text{Mn}_x\text{TiO}_3$ ceramics, ranging from 20 to 35 μm , and for $\text{SrTi}_{1-y}\text{Mn}_y\text{O}_3$ ceramics, ranging from 0.6 to 0.8 μm , without obvious dependence on Mn content. Precursor type (containing Mn^{2+} or Mn^{4+}) had no strong influence on final properties of $\text{Sr}_{1-x}\text{Mn}_x\text{TiO}_3$ ceramics: the lattice parameter keeps the same trend, the average grain size and energy dispersive spectra are similar. Mn solid solubility limit at Sr site of ST determined to be about of 2%. Small grains of MnTiO_3 were found by TEM/EDS to appear in the grain boundaries of $\text{Sr}_{1-x}\text{Mn}_x\text{TiO}_3$ ceramics for higher Mn concentrations. No second phase and strong decrease of the lattice parameter with increasing y were observed for $\text{SrTi}_{1-y}\text{Mn}_y\text{O}_3$ ceramics. These facts point to the incorporation of Mn into the perovskite lattice in the whole experimental range of concentrations $0 \leq y \leq 0.1$. Additional lines in Raman spectra of SMnT and STMn ceramics imply the formation of the additional structural bonds with own dynamics, also confirming the incorporation of Mn into the Sr- and Ti- sites of the perovskite lattice respectively. Raman spectra are different, depending on the lattice site substitution and Mn content. Polar behavior was found in $\text{Sr}_{1-x}\text{Mn}_x\text{TiO}_3$ ($x = 0.005\text{-}0.02$) ceramic system. Radio frequency dielectric measurements showed a diffuse maximum at 25-65 K shifting to higher temperatures with increasing measurement frequency and amount of Mn. The observation of hysteretic behavior in the P vs E curves evidences the existence of a polar state at low temperatures. The hysteresis response slowly degenerates into just nonlinearity as the temperature increases. The observed dipole-glass-type dielectric behavior is attributed to the formation of electric dipoles and corresponding random fields due to off-center position of Mn^{2+} ion at Sr site of highly polarizable SrTiO_3 lattice. It is considered also, that due to the formation of polar microregions, related to the introduction of Mn^{2+} into the Sr site, the temperature range, where the dielectric constant is tunable, was enlarged up to ~150 K, comparing with undoped ST (~80 K). As well, a higher tunability (~70%) and quality factor of a tunable component (~6000) in the vicinity of liquid nitrogen temperature were obtained for SMnT ceramics. Hence $\text{Sr}_{1-x}\text{Mn}_x\text{TiO}_3$ is a promising material for possible application as a phase shifter. On the contrary, $\text{SrTi}_{1-y}\text{Mn}_y\text{O}_3$ ceramics prepared by conventional mixed oxide method, reveal just a sharp reduction of the dielectric response, without any dielectric anomaly. Although incorporation of Mn^{4+} into Ti site of ST lattice, decreasing fractional tolerance factor t_2 , indicates smaller B-site packing degree and supposedly more favorable off-centre positions of these ions, the quantum fluctuations became more stable and the system is driven away from ferroelectricity, corroborating the fact that sharp compression of the cell of STMn system with increasing Mn content does not allow the Mn^{4+} ions to occupy the off-centre position at Ti site as well as less polarisable Mn^{4+} ions substitutes more polarisable Ti^{4+} ions, breaking also Ti-Ti long-range interaction.

Nonstoichiometric strontium titanate (ST) ceramics with precisely controlled Sr/Ti ratio of 0.997-1.02 were synthesised by solid state reaction. Structural properties and microstructure development was examined by XRD and SEM. The dielectric properties were evaluated as a function of the temperature and frequency in the radio frequency range. The effect of Sr/Ti ratio

(1.02 - 0.997) on the grain size and dielectric response was analysed in ST ceramics and preliminary results were reported. No second phases were detected for the studied samples. SrO excess impedes the grain growth and thus decrease the dielectric permittivity. In contrast, TiO₂ excess promotes the increase of the dielectric permittivity values. The variation of Sr/Ti ratio has no strong effect on the quantum paraelectric behaviour of ST and no dielectric anomaly was observed. More detailed analysis showed that Sr_{0.997}TiO_{2.997} can be used as high dielectric permittivity composition and Sr_{1.01}TiO_{3.01} as low loss material.

The solid solubility limit of atomic substitutions is expected to be dependent on the size scale (bulk ceramics and films) of materials and on processing. In this work the incorporation of Mg on A and B sites of SrTiO₃ (ST) thin films prepared by sol-gel on Pt/TiO₂/SiO₂/Si substrates was investigated. The solid solubility of Mg in ST thin films is limited and depends on the lattice site occupancy as observed for identical ceramic compositions. However the solid solubility limit of Mg in ST films (A and B site) prepared by sol gel is higher, than in ST ceramics. Due to a high degree of homogeneity at the molecular level attained in the sol-gel precursor and to the thermal strains induced in the film by the underlayer Si substrate, a higher atomic substitution is tolerated in film's lattice structure. The solid solubility of Mg evaluated by XRD analysis, is limited to $x \leq 30\%$ for A-site occupancy (SMT) and to $y \leq 40\%$ for B-site occupancy (STM). It is observed that the lattice constant decreases for SMT and increases for STM films with increasing concentration of Mg. The dielectric constant and loss tangent at room temperature are reduced for SMT and STM films. The losses of Mg doped ST films are lower than 0.05. The lower polarizability of Mg ions and the tensile thermal stresses induced in the film can account for the observed decrease of ϵ_r and $\tan\delta$ with increasing Mg content. On the other hand the increase of tunability of SMT films at room temperature not observed in equivalent ceramic compositions points to the modification of ST lattice polarization. The tunability of doped ST films is about 6% at 100kV/cm² and room temperature and its dependence on the Mg contents varies with dopant lattice site occupancy. The substitution of Sr or Ti by Zn in SrTiO₃ thin film was also studied. Crystalline Sr_{1-x}Zn_xTiO₃ (SZT) and SrTi_{1-x}Zn_xO₃ (STZ) films (with $x = 0, 0.01, 0.05, \text{ and } 0.10$) having perovskite structure were fabricated on Pt/TiO₂/SiO₂/Si substrates by sol-gel. No second phases were detected within the sensitivity of high-resolution XRD for any of the prepared films. The lattice constant increases for A and B-site doped ST films. The typical dielectric constant and dissipation factor of undoped SrTiO₃ films measured at room temperature and at a frequency of 10 kHz is 225 and 0.05, respectively. The permittivities for ST films decrease with Zn increasing. The loss tangent for Zn doped ST films for A and B-site are about 0.05 at 10 kHz. Capacitance of SZT and STZ films shows small variation with the applied voltage. The effects of Zn doping are a significant reduction of the dielectric constant, dielectric losses, tunability and capacitance compared with pure SrTiO₃ films.

Nanoscale Properties of Ferroelectrics. The investigations into the local properties of ferroelectric films and ceramics continued. The local phenomena were studied in PZT films of different compositions, in ultrathin SBT films and commercial PZT ceramics (PIC151). The microstructure-local property-macroscopic property relationships were established in PZT films of different compositions and thicknesses. It was found that the major parameters governing domain populations in PZT films

are the texture and grain size. It was observed that the thickness of the films does not have any significant effect on the local response, which is sensitive only to the surface layer. It was shown that the SBT films of 40 nm in thickness still exhibit a strong ferroelectricity at the nanoscale level, and the orientation of the grains is a major factor limiting ferroelectric and piezoelectric properties in this anisotropic material. We also studied ferroelectric domains in high-quality SBT single crystals by means of SFM and optical techniques and could explain the appearance of ferroelastic twins by the high temperature ferroelastic-paraelectric phase transition. The study of the local poling of bulk ferroelectrics revealed that, under some conditions, inverse switching can be observed and this switching is related to the injection of charge carriers and electric field reversal. Anomalous switching is shown to be advantageous for the information data storage since the size of the artificial domain can be smaller than 20 nm.

The study of the nanoscale properties of relaxors was extended to the ceramics PLZT, PMN-PT, and BaTiO₃ doped with La(Mg,Ti)O₃ – BT-LMT. In the former case, a local ordering was observed within individual grains and the correlation length was directly determined from the images. The local order was found to depend on the orientation of the grains. It was shown that mechanical stress does not have an effect on the disruption of the ferroelectric state in PLZT. In PMN-PT and BT-LMT ceramics we could follow the phase transition into the paraelectric phase and observe the disappearance of polar clusters. The existence of the structural phase transition between the rhombohedral and tetragonal phases was directly confirmed by SFM. Pure PMN films were investigated by SFM and local phase transition has been observed under a small voltage applied to the SFM tip. The local ferroelectric phase can be stable for the long time. It was demonstrated for the first time that the phase diagram of the relaxors in the surface layer is different from that of the bulk.

The study of the nature of giant piezoelectric response in PZN-PT single crystals was extended to the new compositions: 0.1PbZn_{1/3}Nb_{2/3}O₃-0.09PbTiO₃ and 0.33PbMg_{1/3}Nb_{2/3}O₃-0.67PbTiO₃ which are closer to the morphotropic phase boundary. It was confirmed that the nanoscale domains play an important role in the high piezoelectric response. Various domain patterns were visualized and analyzed by statistical techniques.

The commercial PZT ceramics was investigated at different fatigue stages. It was shown that fatigue mainly occurs near the electrode and after polishing of 100 μm the fatigue-induced modification of the domain structure disappears completely. A model of fatigue in ceramic materials and nature of the defects responsible was proposed.

Bi₂Sr₂CaCu₂O_{8+x} single crystals grown by the self-flux method were investigated using X-ray diffraction, SEM and SFM. The study revealed the existence of incommensurate modulation. Surface morphology was characterized in terms of growth steps, slip lines/bands and etch pits. The observed surface features revealed that the crystals grew by two-dimensional nucleation and etch pits were created by the atmospheric attack. The incommensurate modulation was analyzed and explained. Roughness in GaN/InGaN thin films was studied by Rutherford backscattering and SFM. The RBS results were compared with SFM and excellent agreement was found between the two techniques. In GaN/InGaN multiple quantum wells the roughness was studied as a function of the composition and number of wells grown in stack.

Novel Characterization Techniques. A method of piezoelectric measurements of thick film and bulk samples by a commercially available Fotonic Sensor™ was developed. The main problem of using this technique is a high requirement to the electrode quality and an overestimation of the piezoelectric coefficients due to bending effects. Both problems were successfully solved by using special mechanical interface (cantilever beam) that transfers mechanical displacement from the moving piezoelectric surface to the mirror. The capabilities of the piezoelectric measurement setup were validated by the measurements in both bulk ceramics and thick Pb(Zr,Ti)O₃ films. The technique is viable for the displacement measurements down to 4 Å.

MAGNETOSTRUCTURAL MODULATION OF STRONGLY CORRELATED ELECTRIC MATERIALS

Colossal Magnetoresistive Materials. A main topic of study is the complex interplay of lattice structure, oxygen vacancy, defects and doping on the properties of CMR manganites. This leads to phase segregation at different length scales: charge or orbital ordered; insulator vs metallic. There is prospect of application to magnetic cooling using the magnetocaloric effect.

Preparation of bulk and thin film samples: a) La-(Ca,Sr)MnO₃ and rare-earth (Er,Eu) doped b) Pr-CaMnO₃ system and derived with vacancies in A and B site: Influence of substrate induced strains on the (magnetic and electric) properties.

Structural studies (X-ray diffraction) for phase purity, lattice parameters. Temperature study of structural phase transitions

Magnetic studies: Physical properties in the vicinity of phase transitions. Application of Landau theory of phase transitions to provide a systematic understanding. Magnetostructural coupling.

Magnetocaloric properties: effect of RE substitution on the cooling power for near-room-temperature applications. Study of magnetic entropy in competing phase systems (Ferromagnetic and charge-order)

Electrical properties: electrical resistivity and magnetoresistance in mixed phase regions. Non linear effects and exchange bias.

Hyperfine local probe using implanted radioactive isotopes at ISOLDE-CERN, with Perturbed Angular Correlation Spectroscopy and Emission Channeling: to provide local and element selective information on doping mechanisms. Lattice site and electronic characterisation of the doping elements, disorder and quenched random field effects at the Mn site, in the vicinity of the charge or orbital ordered/ferromagnetic phase instability. Polaron dynamics and percolative effects in lightly doped ferromagnetic insulator manganites.

Theoretical approaches to magnetic materials using generalized thermodynamics.

Description of complex systems with long range interactions or distributed characteristic parameters using Tsallis non-extensive statistics.

High-Temperature Superconductors. Magnetic studies of oxide superconductor materials prepared by LFZ: phase studies, critical current and their relation with structural and phase characteristics in BSCCO fibers. Studies in new MgB₂-type superconductors: preparation and HIP processing. Magnetic and electrical properties studies: superconducting fraction and critical currents. Hyperfine studies on Hg-HighT_c superconductors. Role of oxygen defects in fluorinated compounds.

ADVANCED MOLECULAR AND SUPRAMOLECULAR MATERIALS

Hydrogen Bonds. The main objective of this project is the study of C-H...O hydrogen bonds and their role in the formation of supramolecular structures, from simple dimers to large molecular aggregates. The importance of C-H...O hydrogen bonds in the molecular association has been assessed for a group of carbonyl containing systems, using both theoretical (*ab initio* calculations) and experimental techniques (vibrational spectroscopy, NMR spectroscopy, inelastic neutron scattering and X-ray crystallography). A milestone for this work was the development of some concepts towards the understanding of the unconventional spectroscopic behaviour of C-H...O bonded systems

Cyclodextrins. NMR studies of the cyclodextrin/decanoic (or hexanoic) acid systems in the presence of various anions of group 1 halides have been used to determine the conformation of the guest molecule, equilibrium constants and aggregation properties of decanoic/hexanoic acid included in various cyclodextrins (α , β and trimethyl).

Oxomolybdenum Catalysts. In recent years, complexes of the type $\text{MoO}(\text{O}_2)_2(\text{L}_1)(\text{L}_2)$ and $\text{MoO}_2\text{X}_2(\text{L}_1)(\text{L}_2)$ ($\text{X} = \text{Cl}, \text{Br}, \text{CH}_3$), with different combinations of base ligands L_1 and L_2 , have been experimentally characterized and tested as catalysts for epoxidation reactions, usually employing *tert*-butyl hydroperoxide (TBHP) as the mono-oxygen source. Studies on the mechanism of the epoxidation catalysis with $\text{MoO}_2\text{X}_2\text{L}$ as the catalyst precursor and TBHP as oxidizing agent have been ongoing. A kinetic model of the epoxidation reaction using the model compounds $\text{MoO}_2\text{Cl}_2[p\text{-tolyl-2,3-dimethyl-1,4-diazabutadiene}]$ and $\text{MoO}_2(\text{CH}_3)_2[p\text{-tolyl-2,3-dimethyl-1,4-diazabutadiene}]$ was proposed and supported by UV/Vis spectroscopy and GC/MS. We have found that the catalyst formation may not be a rate-limiting step of the multiple reaction mechanism, which differs from previous findings on tungsten complexes and with theoretical investigations on Mo complexes in which it was suggested that the first step (catalyst formation) is the rate-determining step. We have been particularly interested in developing heterogeneous catalysts based on these complexes, since these can be easily separated from a reaction mixture and recycled, which is of significant industrial interest. The immobilization of homogeneous catalysts on solid supports generally requires additional modification of the catalyst, frequently leads to partial loss of activity, sometimes due to metal leaching from the solid into solution, making them unattractive for industrial applications. A promising alternative is to immobilize the catalyst in a room temperature ionic liquid (RTIL), *i.e.* an organic salt with a melting point below ambient temperature. A series of RTILs were tested as solvents for neutral $\text{MoO}_2\text{Cl}_2\text{L}$ and cationic $[\text{MoO}_2\text{CIL}']\text{BF}_4$ complexes in the catalytic epoxidation of *cis*-cyclooctene, using TBHP as the mono-oxygen source. In general, the best results were obtained using the RTIL 1-*n*-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ([BMIM]NTf₂). A novel cationic complex $[\text{MoO}_2\text{Cl}(\text{Bn}_3\text{Me}_3\text{-tame})]\text{BF}_4$ [$\text{Bn}_3\text{Me}_3\text{-tame} = \text{N,N',N''}$ -tribenzyl-1,1,1-tris(methylaminomethyl)ethane] and [BMIM]NTf₂ gave the best results in terms of catalyst/solvent recycling.

Novel Transition Metal Complexes. The synthesis and characterisation of novel polypyridyl ruthenium complexes was continued and expanded to include supramolecular squares of the type $[(Ru(9[ane]S_3)(4,4-bpy)_2)(Pt(bpy))_2]^{8+}$, for use as light absorbers, photoluminescent sensors, and intramolecular energy/electron transfer agents.

To date, catalytic processes using transition metals bound to chiral ligands are essentially dominated by phosphine complexes. The main achievement during 2004 was the isolation of several compounds which help to fill this gap in the family of catalysts. Indeed, we have shown that when ruthenium-arene complexes [with C_6H_6 (benzene) or $C_{10}H_{14}$ (p-cymene)] react with chiral diamines [$C_7H_{16}N_2$ = (S)-1-ethyl-2-aminomethylpyrrolidine, $C_9H_{18}N_2$ = (S)-(+)-2-(pyrrolidinylmethyl)pyrrolidine, or $C_{14}H_{16}N_2$ = (1R,2R)-(+)-1,2-diphenylethylenediamine], five complex cations are formed, $[Ru(C_7H_{16}N_2)(C_{10}H_{14})Cl]^+$, $[Ru(C_7H_{16}N_2)(C_6H_6)Cl]^+$, $[Ru(C_9H_{18}N_2)(C_6H_6)Cl]^+$, $[Ru(C_9H_{18}N_2)(C_{10}H_{14})Cl]^+$ and $[Ru(C_{14}H_{16}N_2)(C_{10}H_{14})Cl]^+$, which were isolated and characterised structurally as Cl salts using single-crystal X-ray diffraction studies. These complexes exhibit catalytic activity in the transfer hydrogenation of acetophenone to 1-phenylethanol. Another milestone was the use of the Schiff base 2,2'-bis((4S)-4-benzyl-2-oxazoline) which, with rhodium(I) and palladium(II) complexes containing 1,5-cyclooctadiene and/or allyl ligands, led to the isolation of two promising materials, $[Rh(C_{20}H_{20}N_2O_2)(C_8H_{12})][Rh_2(C_{20}H_{20}N_2O_2)_2]^{-}(CF_3SO_3)_3 \cdot (CH_3CH_2O)$ and $[Pd(C_{20}H_{20}N_2O_2)(C_3H_5)]CF_3SO_3$.

Bi- and/or polimetallic (homo or heterometallic) complexes: synthesis and characterization of complexes in which metal centres interact through polypyridylic or O-donors type bridging ligands, such as tppz, 2,3-dpp bptz, 4,4'-bipy, 4,7-phen and terephthalate, isophthalate and isonicotinate anions.

Complexes for DNA Probes. The model complex $[(Ru(9[ane]S_3)Cl(9-Et-guanine))]^{2+}$, for the interaction of transition metal complexes with CT-DNA has been synthesized and characterized by NMR and UV/Vis spectroscopy. The interaction of selected complexes with the Fe transport protein transferrin is being probed using NMR experiments such as NOESY and DOSY.

Macrocycles. The molecular design and synthesis of macrocycles with novel architectures towards to afford suitable sensors for organic substrates was carried out. In addition, these ligands can be also used to encapsulate poisoned heavy atoms, such as Cd, Pb, Hg, UO_2 and lanthanides or two small metal ions (Co, Ni or Cu). The synthesis of new macrocyclic sensors for organic substrates requires previously the molecular design of guests with 3-D disposition suitable for the selective *via* theoretical methods. The molecular modelling has been carried out using several theoretical methods, namely molecular mechanics and dynamics calculations. The thermodynamic proprieties, such as binding selectivity of some macrocyclic receptors, were study in solution, by MM-PBSA methods. A significant amount of new macrocycles with large cavities were synthesised and their coordination behaviour with several metal transition ions evaluated and characterised in solution via different experimental techniques such as NMR and EPR spectroscopy. The structures metal complexes were determined by X-ray diffraction when adequate single crystals were obtained.

Molecular modelling studies on three cryptand-quinone ionophores based on a bis calix[4]arene having two ethyl, propyl or butyl linkages, were undertaken by molecular mechanics and dynamics methods. The binding properties and selectivity relatively a wide range of metal ions (K^+ , Na^+ , Cs^+ and Rb^+) were evaluated. The modeling results found confirmed unequivocally that the metal metal cation is complexed via the equatorial route rather than via vertical central route through the calix[4]arene annulus providing a clear evidence for complexation rate and selectivity of these ligands. The insertion mechanism was study in dmso solution via potential mean force methods. Furthermore, the selective uptake of the metals quoted by the three receptors was also study by thermodynamic integration giving a clear indication that the selective of these macrocycles is also determined by the good match between macrocyclic cavity and the ion size of the metal. The relative Gibbs free energies were calculated and they are consistent with the experimental stability constants.

AREA 2 – Advanced Materials for Industrial Applications

REACTIVE CERAMIC COMPONENTS FOR PROCESS CONTROL

Materials For High-Temperature Electrochemical Applications. Alternative materials have been studied for solid-state electrochemical applications such as fuel cells, electrochemical sensors, oxygen pumping, etc. The main emphasis of research has been on the development on solid electrolytes, and improvement of electrode performance at relatively low temperatures. Laboratory scale oxygen pumps and potentiometric sensors were built, integrated in experimental setups, and are being currently used to adjust and/or monitor experimental conditions in furnaces with controlled atmosphere, or special cells used in electrochemical measurements. A combined theoretical and experimental is being performed to assess errors of potentiometric sensors, and to find criteria to correct or minimize these errors. A laboratory scale fuel cell was built for demonstration.

A variety of prospective solid electrolytes has been evaluated by measuring the oxygen ionic conductivity, electronic transport properties and thermal expansion of materials based on zirconias (YSZ,...), ceria (CGO and CSO), LaGaO₃ perovskites, δ -Bi₂O₃, γ -Bi₄V₂O₁₁, La₂Mo₂O₉, Ln_{10-x}Si₆O₂₆ apatites, Gd₂Ti₂O_{7- δ} pyrochlores, etc. One re-examined the applicability of methods used to evaluate minor electronic conductivity contribution in these oxygen ion conductors.

Apatites La_{9.83-x}Pr_xSi_{4.5}Fe_{1.5}O_{26 $\pm\delta$} , La_{9.83}Si_{4.5}Al_{1.5-y}Fe_yO_{26 $\pm\delta$} , La_{10-x}Si_{6-y}Fe_yO_{26 $\pm\delta$} , La_{7-x}Sr₃Si₆O_{26- δ} and La_{9.83}Si_{4.5}Al_{1.5-y}Fe_yO_{26+ δ} were studied as promising low cost solid electrolytes. Structural features related to the presence of Fe were studied by XRD and Mössbauer spectroscopy. The studies comprised effects of composition changes on sinterability, ionic and electronic transport properties, thermal expansion and stability under strongly reducing conditions, based on the dependence of total conductivity, faradaic efficiency, modified emf measurements and Seebeck coefficient measurements.

Increasing temperature from 973 to 1173 K leads to a substantial increase of the electronic contribution to the total conductivity of undoped lanthanum molybdate and La₂Mo₂O₉-based solid electrolytes, including La₂Mo_{1.7}W_{0.3}O₉, La₂Mo_{1.95}V_{0.05}O₉ and La_{1.7}Bi_{0.3}Mo₂O₉, where the stabilization of β -La₂Mo₂O₉ down to room temperature was confirmed by high-resolution X-ray diffraction and DSC. The ion transference numbers were determined by a modified faradaic efficiency technique, to assess the applicability ranges of these ionic conductors. The results show that these materials can be used as solid electrolytes only under oxidizing conditions and only at temperatures below 1073 K, due to the onset of electronic contributions, limited stability range and also relatively high thermal expansion.

NASICON-type Na₃Si₂Zr_{1.88}Y_{0.12}PO_{11.94} and Na_{3.2}Si_{2.2}Zr_{1.88}Y_{0.12}P_{0.8}O_{11.94} ceramics were studied as potential cationic conductors. Structure refinement and the dependence of electrical conductivity on composition, microstructural features and working conditions were used to assess and interpret the transport properties, and to re-examine their stability under prospective working conditions. Other potential cation conductors were also prepared and studied, namely Na_x(M_yL_{1-y})O₂ (M

= Ni^{2+} , Fe^{3+} ; L = Ti^{4+} , Sb^{5+}). These materials may be affected by sensitivity to air humidity and its dependence on ceramic microstructure.

Electrode kinetics has been studied and correlated to ionic and electronic transport properties of cathode and anodes materials, their defect chemistry, and changes in microstructure. Interesting results were found for a combination of $\text{LNi}_{0.8}\text{Cu}_{0.2}\text{O}_{4+\delta}$ cathode and LSGM electrolyte, with clear correlation between electrocatalytic performance, transport properties and kinetics of surface exchange. A special cellulose-precursor method has been used to obtain nanocrystalline $\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{2-\delta}$ fibers, which were used to prepared SOFC anodes by impregnation with salts of the remaining components. Modifications of electrolyte materials were also explored, namely for ceria electrolytes modified with suitable additives.

Microstructural Effects. Microstructural effects were studied in solid electrolytes in order to suppress the blocking effects of resistive grain boundaries. The grain boundary behaviour of ceria based electrolytes (e.g. CGO and CSO) are studied in detail to understand the changes in grain boundary behaviour exerted by changes in composition and by sintering additives. Changes in grain boundary behaviour may also be used to monitor degradation effects under aggressive atmospheres; this was observed for apatite-type electrolytes under strongly reducing conditions and NASICON-type cationic conductors in wet atmospheres. Other microstructural features might exert different effects on the ionic and electronic conductivities, as found in $\text{Gd}_2\text{Ti}_2\text{O}_{7-\delta}$ based pyrochlores.

Mixed Conducting Materials For Oxygen Separation or Partial Oxidation of Hydrocarbons. Thermodynamic calculations were used to analyse expected equilibrium conditions in methane to syngas conversion, the dependence of conversion and gas composition on working conditions, and stability requirements of membrane materials. Quasi-equilibrium conditions were also predicted for mixed conducting membrane reactors, with tubular reactor and CSTR configurations.

Mixed-conducting $\text{La}_{0.3}\text{Sr}_{0.7}\text{Co}_{0.8}\text{Ga}_{0.2}\text{O}_{3-\delta}$ (LSCG) were found to possess substantial oxygen permeability and assessed as prospective materials for partial oxidation of methane. Studies in fixed bed reactors, and by pulsing CH_4 over LSCG powder showed that these materials promote complete CH_4 oxidation. Partial oxidation, thus, requires reforming catalysts. Results indicate that prevailing mechanisms of oxidation reactions on the surface of LSCG may be associated with weak Co-O bonding, and correlate with oxygen desorption, phase stability and ionic transport.

The oxygen permeability of $\text{La}_2\text{Ni}_{0.9}\text{Co}_{0.1}\text{O}_{4+\delta}$ was re-examined to assess the relative roles of bulk mixed transport and surface exchange. Different powder preparation methods also yield differences in oxygen permeability. The oxidation kinetics was measured in a model membrane reactor under air/ H_2 and air/ CH_4 gradients, with a Pt catalyst. Though $\text{La}_2\text{Ni}_{0.9}\text{Co}_{0.1}\text{O}_{4+\delta}$ may undergo degradation under these severe working conditions, the actual membranes could be operated for relatively long times by using a porous protection barrier against very reducing atmospheres. Catalysts were added to this porous layer, by infiltration and/or by in-situ reduction of selective components of the surface layers. These results show that complete

oxidation also prevails for these membrane materials. Though formation of CO increases with temperature, suitable catalysts are still needed for selective partial oxidation of methane to syngas.

A variety of materials are thus being prepared as characterized as potential catalysts for partial oxidation reactions; this includes oxide materials with incorporation of Ce-, V-, Cr-, Mn- or Fe-oxides, rare earth oxides (e.g. Ce- or Pr-oxides), and also combinations of redox pairs.

Systematic studies of ionic and electronic conductivity, bulk transport mechanisms and surface exchange kinetics of prospective membrane comprised different methods such as the dependence of total conductivity and Seebeck coefficient on working conditions (oxygen partial pressure and temperature) and on composition changes, modified emf or faradaic efficiency measurements, oxygen permeability, and coulometric titration. Defect chemistry was analysed in detail for some of these materials, taking into account the effects of changes with compositions and with working conditions. These studies include the mechanisms of ionic and electronic conductivities, oxygen stoichiometry, ordering/disordering of point defects and other structural factors affecting ionic motion. Other studies of structural effects on properties of mixed conductors include thermal expansion, phase stability domains in wide ranges of working conditions and interactions between different components of composite materials or between different cell materials. Structure Refinements were based mainly on XRD, and Mössbauer spectroscopy for a variety of materials containing Fe. Effects of internal interfaces were studied for composite materials and for other ceramics with significant microstructural effects on the electrical conductivity properties. One found that ionic conduction in oxide composites is mainly dependent on phase interactions and grain-boundary behaviour, due to cation interdiffusion, intermediate phases and/or blocking grain boundaries.

Detailed studies were performed for a variety of materials with the following structure types: LaGaO₃-based perovskite materials, such as (La,Sr,Pr)(Ga,Mg,M)O_{3-δ}, M=Fe,Ni,Co,Cu; La_{0.3}Sr_{0.7}Fe(Al)O_{3-δ} perovskites; La₂Ni_{1-x}M_xO_{4+δ} materials (M=Fe,Ni,Co,Cu; x= 0.1-0.2) with interlayered perovskite/rock salt structure; layered perovskites (TbBaCo_{2-x}Fe_xO_{5+γ}; 0.08 ≤ x ≤ 0.24); strontium ferrites with different structural features ranging from perovskite type SrFeO_{3-δ}, to brownmillerite SrFeO_{2.5+δ}, intergrowth Sr₄Fe₆O_{13+δ}, and Ruddlesden-Popper type Sr₃Fe₂O_{6+δ}; garnets based on gadolinium ferrite such as Gd₃Fe₅O_{12±δ} and Gd_{3-x}M_xFe₅O_{12±δ} with M=Pr, Ca; oxide composites combining one ionic conductor (LSGM or CGO) and one electronic conductor such as La_{0.8}Sr_{0.2}Fe_{0.8}Co_{0.2}O_{3-δ}, La_{0.7}Sr_{0.3}MnO_{3-δ}, La₂Ni_{0.8}Cu_{0.2}O_{4+δ}, SrCoO_{3-δ} or Sr₂Fe₃O_{6.5±δ}.

CERAMIC COMPOSITES AND ULTRA-HARD COATINGS FOR MECHANICAL APPLICATIONS

Diamond Coatings. Si₃N₄ round inserts were direct coated with diamond films using microwave plasma assisted CVD technique. These were tested in turning of hardmetal (WC/25wt%Co) cylindrical bars, in dry environment, varying cutting speed, feed and depth-of-cut. The cutting velocity was optimized to obtain good finishing and to avoid the delamination of the diamond film and tool failure.

The behaviour of CVD diamond brazed tools was investigated in dry turning of sintered hardmetal (WC/25wt%Co). A clear relationship between cutting tool forces, flank tool wear and workpiece finishing was found and used to obtain a good finishing and low tool damage. It was shown that the monitoring of cutting forces during the turning operation assures a good performance of the tool. Damage of cutting tools was caused mainly by flank tool wear, cratering on the rake tool face and hardmetal deposition on the tool surface.

Carbon fibre reinforced carbon composites were turned using uncoated and CVD diamond coated Si_3N_4 cutting tools. Si_3N_4 inserts were fabricated by pressureless sintering and CVD diamond coated. Turning tests performed with uncoated tips showed that cutting forces increase with the cutting speed and cutting length. Severe wear of the tool flank face takes place due to rubbing by the abrasive carbon powder generated during the turning operation, the tool wear increasing with the cutting speed. Conversely, CVD diamond coated Si_3N_4 tips exhibited low and constant cutting forces at different cutting parameters, due to the formation of a carbon lubricant layer.

A home-made HFCVD reactor was used for deposition of nanocrystalline (NCD) films on Si_3N_4 substrates, using $\text{Ar-H}_2\text{-CH}_4$. The surface morphology and quality of the NCD deposits were characterized. The optimal deposition parameters were found on varying the filament temperature, substrate temperature, flow rate, and Ar/H_2 and CH_4/H_2 gas flow ratios.

The incorporation of fluorine in DLC films is promising to modify electrical, optical and tribological characteristics. A nanoindentation technique was used to characterize the mechanical behaviour of very thin F-doped coatings, grown by RF magnetron sputtering. The Vickers nanohardness and the Young's modulus values fall in the 55-70 GPa and 235-326 GPa ranges, respectively.

Diamond films were also deposited on WC-Co hardmetals by time modulated chemical vapour deposition (TMCVD). TMCVD increased drastically the number of diamond crystallites nucleating onto WC-Co, yielding smooth surface profile, improved film coverage and better coating adhesion. The adhesion was characterised by indentation tests, and micro-Raman spectroscopy was used to calculate the biaxial stresses.

Colloidal Processing of Materials. An innovative method was developed for protecting AlN powders against hydrolysis and for preparing stable and high concentrated (≥ 50 -vol.% solids) aqueous suspensions for colloidal processing (slip casting, tape casting, pressure casting, etc.) and granulation of powders for dry pressing. A thermo-chemical surface treatment of AlN powder was used to prevent hydrolysis and allow AlN aqueous processing. AlN powders were treated by dispersing in warm $\text{Al}(\text{H}_2\text{PO}_4)_3$ water solutions. Dense samples were then obtained by sintering at 1750 °C for 2 h. Effective dispersion was achieved with commercial dispersants, and high solids loading. Good dispersion yields green samples with high particle packing and high sintered densities ($>96\%$).

Different de-agglomeration routes were tested for dispersing multicomponent reaction sialon precursor powders and to establish a methodology for preparing homogeneous, highly concentrated suspensions. Reaction sialon suspensions were prepared using 3-wt% KD1 as dispersant in organic media of methylethylketone+ethanol. Different homogenizing

procedures and solid loading were used to study the effects on the rheology of suspensions and on green bodies obtained by slip casting.

Stable and low-viscosity suspensions of SiC, Y₂O₃ and Al₂O₃ powder mixtures with up to 60% vol solids were prepared in methyl ethyl keton (MEK)/Ethanol (E) solvent. The gelation mechanism is mainly based on the collapse of adsorbed layer as temperature decreases, which induces incipient flocculation and form a stiff network. The gelled body was further strengthened by separation of dispersant from suspension, yielding green bodies with uniform microstructure, and highly dense ceramics upon pressureless sintering.

Modification of alumina powders from hydrophilic to hydrophobic was successfully used to assist the colloidal processing of alumina, namely low-pressure injection moulding

A systematic study was performed of effects of particle size, particle size distribution and milling time on the rheological behaviour, particle packing of silica suspensions, and their green body packing.

Cordierite sintered foams were fabricated by polymer foam replication process and firing.

Macroporous anatase membranes were prepared by dip coating via the sol-gel method, using acetic acid as stabiliser and poly(ethylene glycol) as additive. Processing parameters, such as the amount of PEG and withdrawal speed were found to play important roles in the microscopic features of the membranes.

CaMg₃Al₂Si₂O₁₀F₂ mica with incorporation of Li, Na, K and B was used to obtain glass ceramics. These glass-ceramics exhibited capability for easy bulk crystallization, high whiteness, translucency, and mechanical and chemical properties suitable for several applications. Glass-ceramics were also obtained by both bulk crystallization and sintering of glass powder compacts with compositions between tetra-silicic mica and fluorapatite-diopside 50/50 (in wt.%). Lithium aluminosilicate glasses and glass-ceramics were obtained from spodumene-based materials.

Crack-free cordierite-glass tapes were prepared via aqueous tape casting. The influence of solids loading, average particle sizes, binders and plasticizers on processing step has been studied in order to optimise the tape casting process.

A process was developed to convert models made by rapid prototyping into metallic moulds. The effects of ceramic materials (shape, granulometric distribution, chemical composition), sintering (time and temperature) and casting conditions (mould pre-heating temperature and pouring temperature) were studied in order to obtain ceramic moulds and, subsequently, metallic moulds with tailored properties. Doping of aluminosilicate crucibles with MgO, CaO, or BaO was aimed for improving their performance towards Al-alloy melts in aluminium foundry industry.

Industrial wastes were incorporated in alternative recycled materials formulations. Granite wastes were incorporated in the batch formulations of porcelain tiles. Samples of different formulations were produced at both laboratory and pilot-plant scales. These results show that incorporation of granite sludge can result in porcelain tiles with superior properties. Al-rich sludge from anodising and surface treatment was tested in the fabrication of mullite/alumina-based materials. Different compositions were prepared by using sub-products or natural silica-containing materials, like ball clay, kaolin and/or diatomite. Ultimate properties of these materials are dependent on the shaping method and sintering schedule. Residues from pine, blue gum and cork, were used to produce wood-cement composites. The interaction of lignocellulosic materials in the

reaction of cement with water was studied to evaluate its effects on cement setting, thus affecting the properties of the wood-cement composites.

Synthesis and Characterization of Powders. Hydrothermal synthesis was used to obtain titania nanoparticles. TEM revealed that the morphology of the particles formed at low TENOH concentrations consisted of well-dispersed anatase crystals, changing to asterisk-like structured particles with increasing the concentration of TENOH.

Mullite whiskers were produced by firing kaolin at 1400-1550 °C, with CuSO₄ as fluxing agent.

Combustion synthesis was used to prepare powders of technical ceramics. Single-phase ternary carbide of Ti₂AlC_{1-x} was combustion synthesized with nominal composition Ti:Al:C 2:1:0.7. It was found that a low combustion temperature is beneficial to the formation of single phase Ti₂AlC_{1-x}. Rod-like Y stabilised sialon seed crystals were also synthesized by combustion synthesis and used as reinforcing agents of reaction α -sialon ceramic matrixes. The effects of nitrogen pressure and NH₄F additive on combustion synthesis of rod-like α -SiAlON crystals were investigated. The microstructural development of rod-like α -SiAlON crystals revealed anisotropic grain growth with prevailing basal plane growth.

Methods have been successfully developed to modify ceramic powders for suitable wet processing. This includes the application and chemisorption of protective monomolecular layers of long-chain carboxylic acid or its salts were used to modify the surface of alumina powders from hydrophilic to hydrophobic, and dispersion of AlN powders in warm water solutions of Al(H₂PO₄)₃, to enable the processing of covalent ceramic powders in aqueous media.

AREA 3 – Chemistry and Technology of Polymeric and Lignocellulosic Materials and Biomaterials

MACROMOLECULAR MATERIALS AND LIGNOCELLULOSICS

Lignocellulosics. The studies aiming to understand the chemistry of *Eucalyptus globulus* wood during pulping and bleaching and the comparative study with other hardwoods were concluded. The information obtained allowed the clear understanding of the peculiar characteristics and behaviour of *E. globulus* in industrial processes, namely its easier pulping and bleaching.

The investigation of different approaches or process modifications to improve the retention of polysaccharides during *Eucalyptus globulus* kraft pulping was pursued. Different kraft process modifications, including the levelling of the sulphide charge along the pulping process and the addition of borates and amines at different stages of the cooking were investigated. Conditions allowing the higher retention of polysaccharides were identified. Simultaneously, studies aiming to identify pulping conditions that allow the precipitation of black liquor dissolved xylan at fibre surfaces were initiated. The effect pH of black liquor in contact with fibre at the end of the pulping, on the xylan and lignin precipitation was investigated. The characterization of the precipitated xyans was initiated; this included the surface analysis of fibres by XPS and ToF-SIMS.

The application of Py-GC-MS technique in wood characterization was pursued. The aim of this study, carried out in cooperation with RAIZ, is to relate the chemical composition and structure of different *Eucalyptus globulus* clones with their pulping performance. About 50 clone samples were characterized by Py-GC-MS and the lignin syringyl: guaiacyl ratios were determined. Part of such samples was characterized for their monosaccharide composition and lignin content. The GC-MS analysis of the lipophilic extractives was initiated.

Different *Acacia* species (wood and bark), invasive trees in the Portuguese forest, were characterized for their lipophilic components, aiming to identify new constituents with potential bioactivity. New caffeates, sterols and sterylglucosides were identified by GC-MS for the first time in the investigated species. The isolation of some of these compounds, aiming to test their biological activity, was initiated.

The controlled heterogeneous modification of cellulose fibres by esterification with fatty acids of chain length between C6 and C22, using fatty acyl chlorides/pyridine reaction system, was pursued. The use of vegetable and animal oils and waxes as sources of fatty acids was attempted. A new approach to determine the degree of substitution (DS) by alkaline hydrolysis and GC-MS analysis of the liberated fatty acids, was developed. Composite samples were prepared by thermal pressing of the modified thermoplastic fibres (high DS) or by thermal mixing and pressing of fibres (low DS) with polypropylene. The characterization of the composites by DSC and DMA was started.

The preparation of cellulose fibre hybrids with nano-particles was initiated. Different approaches to prepare cellulose/TiO₂ materials were investigated, including sol-gel and controlled hydrolysis of Ti⁴⁺ in acidic conditions. The TiOSO₄/H⁺ system

was selected and optimized. The impact of reaction parameters on the extent of TiO_2 deposition and particle size and morphology was investigated. Hybrids were characterized by ICP, Raman, DRX and SEM. The papermaking properties of a selected cellulose/ TiO_2 hybrid were assessed and compared with samples of blends of cellulose fibre and commercial TiO_2 (mechanically mixed). Paper samples prepared with the hybrid showed much higher opacity than the corresponding blends.

Silica-cellulose hybrid (SCH) materials based on primary sludge and bleached/non-bleached beaten eucalypt kraft pulps were obtained under optimized conditions in a larger scale. The techniques for the preparation of finished material (molds, etc.) were developed. The obtained silica-cellulose materials were tested on water resistance, thermal degradation and thermal conductivity. Thermal analysis showed an increase in thermal resistance of the hybrids. Additionally, CSH showed much higher hydrophobicity than the starting cellulose material. It was also observed that varying the synthesis conditions ($\text{H}_2\text{O}/\text{TEOS}$ ratio, EtOH/TEOS ratio and pH, etc.), CSH with different molecular structure might be obtained. The preliminary results on thermal conductivity indicated CSH as a perspective thermal insulation material.

Exploratory experiments aiming to evaluate the performance of cellulose or cork with phase change materials for insulation and energy storage applications were initiated. Commercial phase change materials were characterized and composite materials, including polymer mortars, were prepared for mechanical and thermal testing.

The work on polyoxometalate (POM) catalysis in oxygen delignification using combinatory (alteration treatment of unbleached kraft pulp) approach with laccase in a multi-stage system for the oxygen bleaching has been accomplished. A multi-stage system using robust heteropolyanion $[\text{SiW}_{11}\text{Mn}^{\text{III}}(\text{H}_2\text{O})\text{O}_{39}]^{5-}$ and laccase of *Trametes versicolor* for the oxygen bleaching of eucalypt kraft pulp was developed. This combines the activity of $\text{SiW}_{11}\text{Mn}^{\text{III}}$ as selective catalyst for the residual lignin oxidation with the laccase capacity to re-oxidise the heteropolyanion hardly reacting with oxygen even at vigorous conditions. Such an approach allowed more than 50% of kraft pulp delignification with minimal (around 5%) polysaccharides damage. Being stable under reaction conditions, the catalyst $\text{SiW}_{11}\text{Mn}^{\text{III}}$ can be continuously re-used in the treatments. Several approaches on the development of the continuous bleaching process have been proposed.

A series of polyoxometalates ($\text{PMo}_{12-n}\text{V}_n$ ($n=2,4$), $\text{PW}_{12-n}\text{W}_n$ ($n=4,6$), $\text{SiW}_{12-n}\text{Mn}_n$ ($n=1,2$), W_{12}Co , SiW_9Ru_4 between others) were deposited on the modified (aminated)/non-modified cellulose-silica hybrid aiming to use it like support in the catalytic dehydrogenative oxidation of ethylbenzene in styrene in heterogeneous catalysis with oxygen at 320°C . The best selectivity was obtained with SiW_9Ru_4 though with moderate yields (10-15%).

The study on the nature of lignin-carbohydrate complexes (LCC) in the eucalypt wood was continued. In order to understand better the NMR data obtained previously on ^{13}C -labeled LCC, a series of LCC model compounds containing phenyl glycoside and benzyl ether type linkages were synthesized and thoroughly characterised. This allowed the identification the corresponding structures in wood tissues.

The investigations on ESI-MS application for the structural characterization of lignin were continued. Using a database obtained from analyses on lignin model compounds, an effort was done to develop a methodology for the elucidation of lignin oligomers structure from different morphological regions. This allowed proposing a model structure of the lignin in the secondary cell wall.

The data obtained previously on muconic acid model compound were extrapolated to the oxidized lignin samples aiming to create a reliable methodology for the detection of muconic acid type structures applying 2D NMR techniques. The reliable identification of muconic acid structures in oxidised lignins was proposed.

A project on the improvement of the yield and the mechanical strength of acid sulfite pulp was started. The main idea was to optimize the pulping conditions. At the first step of this investigation the base concentration in the pulping acid was increased from 30% to 2-3 times and the effect of changes made in pulping on the pulp yield, mechanical strength properties and the bleachability have been estimate. The pulping conditions were optimized in the manner to maintain the total pulping time the same.

The project on the improvement of sulphite pulp brightness in P(O)-P bleaching was started. The main idea was to optimize the bleaching conditions in order to increase the pulp brightness from actual 88-89% ISO to 90 -91% ISO. Additionally, different hydrogen peroxide activators/catalysts were employed on the second bleaching stage. This yielded around 90% brightness. Most effective is the application of the bleached pulp treatment with reducing reagents. The best result was obtained with sodium dithionite salt allowing to reach 91% brightness. The results of laboratory experiments were completely confirmed in the industrial assay carried out on the installations of Caima Cellulose Company.

Other Polymer Systems and Materials. Significant progress was made in connection with the preparation of polymer based nanocomposites (NC) *via* ATRP. Q-dots@TOPO/PBA NC have been prepared by miniemulsion. These materials show novel optical properties.

In September we started investigating the polymer@SiO₂ nanoparticle interfaces. This work is helping to tune surface modification of SiO₂ and thus improve the preparation of silica NC based on synthetic polymers and on polysaccharides. We are also performing zeta potential and light scattering measurements. A PhD programme focuses on the preparation and characterisation of SiO₂/exopolysaccharide NC.

The flexibilization of a phenolic resin for use in the production of sandpaper, in collaboration with a local industry, was achieved.

Polymer (amorphous and semi-crystalline) materials quantitative experimental creep and theoretical non-simulative molecular dynamics detailed studies were finalized and a Ph. D. thesis successfully concluded, with the development of a predictive non-linear creep model.

The work on thermal properties and crystallization of polymers, including developments on the use of DSC measurement techniques, was pursued.

A predictive, analytical (non-simulative) cooperative segmental theory of molecular dynamics (CSTMD) was developed and extended to all types of thermal (heating/cooling scans) and forced physical excitations. Its initial evaluation already led to a comprehensive explanation of the crossover and glass transition behaviour and to significant insight into the origin of cooperativity and its dependence on temperature and time-scale (temperature scanning rate or forced frequency) of the excitation /observation.

BIOMEDICAL AND BIOMIMETIC MATERIALS

Biological, Structural and Identification FTIR, NMR and Other Studies. New Cr(V) compounds, with ligands biologically relevant, such as quinic acid and small peptides, were synthesised and characterised. These new compounds have been tested in *in vivo* with mice submitted to severe or chronic intoxication. Target organs have been checked for degenerative damages by histological, histochemical and ultra-structural methods in order to evaluate and to clarify chromium reduction intermediates toxicity mechanisms. The effects of some ions released from biomaterials such as titanium and Cr-Co-Mo based alloys were investigated in mice using cryogenic sections of relevant organs such as the spleen, the kidney, and the liver. Histochemistry for proteins and carbohydrates were also used.

NMR relaxation studies of novel biomedical blended chitosan/soya membranes have indicated that, independent of the preparation conditions, the components are not completely miscible due to a weak polymer-protein interaction. The incorporation of soya in chitosan membranes and cross linking with GA had a clear positive effect on their *in vitro* biological behaviour.

The Ni, Zn and native Fe forms of Desulforedoxin (small model protein) and 3 mutants have been studied by NMR to probe the interactions that stabilise inorganic metal centres in proteins. A Gln side chain H-bond to the metal centre was found to be important for stabilization. TROSY type NMR experiments on $^2\text{H}/^{15}\text{N}$ labelled ALAS at 800MHz indicated the protein to be unstructured under the conditions used. The secondary structure of a related protein, Heme Binding Protein with no known structure, has been determined.

High resolution (HR) MAS ^1H NMR spectroscopy showed promising results for evaluating the quality of donor livers for grafts and study their performance post-graft through characterisation of their metabolic composition. Biopsies from six donor livers were analysed at three transplantation stages, and graft quality could be related to both graft chemical composition and dynamics.

Diffusion-ordered spectroscopy (DOSY) was used in the traditional form and in the form of new experiments to characterise complex mixtures such as beer and port wine to increasing levels of sensitivity and resolution. In tandem with chemometrics, NMR was used to distinguish beers from different origins.

The study of systems in the solid state comprised a detailed molecular dynamics characterisation of several sugars in different physical states (amorphous, crystalline and glassy).

Spectroscopic/chemometrics studies of several flours was performed using FTIR and Raman in order to enable information on flour origin and quality to be obtained. Such aim has proved difficult to achieve and construction of models is being made to include rheological results obtained for the same samples.

Glass and Ceramic-Based Biomaterials. The role of albumin in the mineralisation process of commercial hydroxyapatite and synthesized biphasic (HAP-TCP) ceramics in a buffered SBF by CO_2 / HCO_3^- was studied. An appropriate gas mixture of

CO₂/N₂ was continuously bubbled in SBF solution without Tris / HCl. This method renders possible to buffer the solution in the 7.3 – 7.4 pH interval and to reach a HCO₃⁻ concentration between 24 and 27 mmol.dm⁻³, which are the normal concentration values in the blood plasma. The results suggest that mineralisation studies in this modified SBF mimetic biomineralization more closely than traditional SBF.

The influence of solution composition (pH, presence of Na, K, Mg and Cl ions) and temperature on the crystallization and ageing of the calcium phosphate solid phases was investigated and the aqueous solutions were studied by ICP. The composition of the solid phases was determined by wet analysis and by XRD, TG and IR.

Glass compositions have been obtained from the three systems SiO₂-CaO-P₂O₅-MgO, SiO₂-P₂O₅-CaO-MgO-K₂O and SiO₂-Na₂O-MgO. Particular glass compositions were submitted to adequate heat treatment schedules to obtain glass-ceramics. The undertaken research had two main objectives: (1) to study the relationship between structure and surface reactivity of silica-based glasses and glass-ceramics, the latter being physically expressed by the apatite layers precipitated on their surfaces after immersion in simulated plasma. Special attention was given to the structural role of modifying oxides; (2) to understand the differences between mechanisms of adhesion of apatite layers to glasses and to glass-ceramics by appealing parameters such as composition, crystallinity, surface topography.

Structural interpretation was based on spectroscopic techniques, such as FTIR, Raman and NMR and surface reactivity was assessed by *in vitro* bioactivity tests in synthetic plasmatic fluids. Surface modifications were followed by SEM observation of samples.

Glasses of the SiO₂-CaO-P₂O₅-MgO system with *in vitro* bioactive behaviour have been added to biodegradable matrices to obtain novel biodegradable composites. The new formulations have been studied in terms of chemical and mechanical behaviour. The results of this research are being collected and discussed in a PhD thesis that is being written.

The preparation of CaP particles with a wide range of sizes by precipitation in calcium/citrate/ phosphate solutions has been addressed. The manipulation of experimental conditions allowed to produce either nanosized particles, either micrometric sized aggregates with particular shapes. Porous calcium phosphate granules were produced by spray drying suspensions of nanosized calcium phosphate particles. Porous granules were characterized by SEM, XRD, FTIR and N₂ adsorption. The prepared granules show the capability to adsorb and release a model drug compound (salicylic acid) in solutions at physiological pH.

Porous chitosan scaffolds were produced by a freezing drying process of chitosan acetic acid solutions. The solution experimental conditions, including the chitosan and acetic acid concentration, were investigated so as to obtain 3D porous scaffolds with 90% of porosity. The bioactivity of the obtained polymeric scaffolds was also studied using ageing procedures in SBF (simulated body fluid) solutions.

New composites that consist of poly(methylmethacrylate)-co-(ethyl-hexyl-acrylate) (PMMA-co-EHA) filled with a glass-ceramic (mol%) (70 SiO₂ – 30 CaO) have been developed. The *in vitro* bioactivity was assessed by determining the changes in surface morphology and composition after soaking in simulated body fluid (SBF) for periods of up to 21 days at 37° C. X-ray diffraction (XRD) and scanning electron microscopy coupled with X-ray energy dispersive spectroscopy (SEM-EDS) after

different soaking periods confirmed the growth of apatite-like deposits after 3 days. The deposits consisted of spherical aggregates of acicular crystallites.

Two ceramics: a $25\text{CaO}-2.5\text{P}_2\text{O}_5-72.5\text{SiO}_2$ mol% sol-gel glass and a commercial hydroxyapatite (HAP) were studied according to two recently proposed in vitro protocols. The first one (SBF-dynamic) avoids the variations of the ionic concentration in the assay solution with a continuous renewal of SBF. The second protocol, uses a carbonated simulated inorganic plasma (CSIP) whereby a physiological concentration of HCO in solution is reached (24 to 27 mM) whereas the pH is maintained between 7.3 and 7.4. In CSIP, a higher HCA crystallization rate was initially observed. Nevertheless, after 7 days in SBF-dynamic, the new layer was thicker and presented a Ca/P molar ratio lower than in CSIP (1.7 vs 2.1). In the case of HAP ceramic, a HCA layer was observed only in CSIP.

Albumin-containing CSIP (CSIPA) was used for mineralisation of two calcium phosphate ceramics and a bioglass of the Si-Ca-Mg-P-O system. It was found that formation of apatite deposits in CSIPA is enhanced when compared to CSIP and also to the classical SBF medium. The results suggest that CSIPA simulates in vivo mineralisation more closely than SBF and also that it may be a suitable medium for biomimetic deposition of apatite.

PROCESS DEVELOPMENT AND OPTIMISATION

Phase Behaviour and Transport Properties Relevant in Environmental Protection, Chemical Processing and New Materials Production. The solubility of oxygen in several liquid perfluorocarbons, perfluoro-n-hexane, perfluoro-n-heptane, perfluoro-n-octane, perfluoro-n-nonane and perfluorodecalin, was measured in the temperature range between 288 and 313 K and at pressures close to atmospheric. Values were measured with an apparatus based on the saturation method with an accuracy of $\pm 1\%$. Thermodynamic functions such as Gibbs Energy, Enthalpy and Entropy of solution were obtained from the composition and temperature dependence.

The validity of a molecular model within a SAFT context for quantitatively predicting the solubility of xenon and oxygen in n-perfluoroalkanes was studied. All species are treated as Lennard-Jones cores or chains formed by tangentially bonded spheres with the same diameter and dispersive energy. Optimized meaningful values of both molecular parameters for the pure perfluoroalkanes are also used to accurately predict vapor-liquid equilibria of n-alkane + n-perfluoroalkane mixtures. Due to the high non-ideality of the mixtures, the Lorentz-Berthelot cross-interaction parameters need to be adjusted using experimental data and ensuring coherent trends. An accurate description of the solubility of oxygen requires additional information to be included in the model. On the basis of ab initio arguments, we considered cross-association between oxygen and perfluoroalkane molecules, which allows describing solubilities with a deviation below 5%, when compared to experimental data available in the literature and measured in our laboratory.

Studies of the properties of long chain alkanes and its description using Corresponding States Theory and Gradient Theory for the interfacial tensions were continued. New studies of crystallization of paraffins from solution were carried for simple

systems aiming at improving the existing models. A new approach to the studies of paraffins was started aiming at developing composite material for thermal insulation.

The solubility of nitrogen, oxygen, carbon dioxide, and water in the vapor phase in PolyLactic Acid (PLA) was measured, using a Quartz Crystal Microbalance, specially built for that purpose. This apparatus was calibrated with the system carbon dioxide in atactic polystyrene and the determined precision was of 1.5 %. Measurements were performed up to atmospheric pressure and in the temperature region from 293 to 313 K. The experimental results obtained were compared with the Flory-Huggins model.

A new activity coefficient model was proposed for correlation of Vapor-Liquid Equilibrium (VLE) of polymer solutions. This model uses a segment based UNIQUAC term, sUNIQUAC, in the residual term and three combinatorial terms were tested, the Entropic free volume, p-free volume and Freed FH. Comparison of the performances of all the possible models that can be obtained based on the best and most used combinatorial (free volume) and residual terms, NRF, UNIQUAC and Wu, was done. A database of 70 VLE data systems was used to evaluate the different models. It is shown that models based on the p-free volume combinatorial term coupled with a segment based NRTL or UNIQUAC residual part can provide an excellent correlation of VLE data for polymer systems and describe a wide range of molecular weights using a single pair of interaction parameters. The NRF based models are shown to suffer from some deficiencies that limit their use in concentrated solutions.

Two studies concerned the treatment of effluents using bioreactors. One addresses the denitrification of effluents and the influence of the C/N ratio on the composition of the microorganisms colonies there present and another aims at using a yeast that has been widely studied in our group towards the decolorization of dyes from textile industry effluents. A third line addresses the increase of production in bioreactors by enhancing the aeration. This has been attempted in two ways: one by increasing the gas pressure on the bioreactor and studies of the influence of the pressure on the microorganisms have been carried; the other uses a second liquid phase to enhance the oxygen availability to the microorganisms, the organic phase we have been studying are the perfluorocarbon compounds that we have been studying.

On modeling and simulation of cyclic separation processes using parallel computing, a low cost 8-PC/Cluster was assembled for carrying parallel computing calculations, especially oriented to the solution of differential/algebraic systems of equations. This type of numerical problem appears normally after the discretization of spatial coordinates of partial differential equations, i.e. mass, energy or moment balance equations inside a separation equipment. The target of this project is the modeling and simulation of a Pressure Swing Adsorption process for producing oxygen. In order to reduce the computing time, parallel computing numerical techniques are being implemented and results compared with ordinary sequential solution of the mathematical model. A tested model has been selected from literature and solved with commercial software. Those results will be reproduced using a parallel numerical alternative with the new cluster just installed.

The conversion of xylose to furfural, in the presence of mesoporous solid acids, has been studied. Furfural is a key derivative, readily accessible from renewable biomass and agricultural surpluses, for the production of a wide range of important non-petroleum-derived chemicals. Furfural can be produced from agricultural raw materials rich in pentosan polymers by acidic degradation, which involves hydrolysis of the pentosan into pentoses (e.g., xylose) and successive dehydration of the latter to

form furfural. Surfactant-templated micro-mesoporous silicas possessing sulfonic acid groups have been prepared, characterized and tested as catalysts in the dehydration of D-xylose to furfural. Sulfonic acid-anchored MCM-41 has been found to be an effective catalyst for the dehydration of D-xylose to furfural. Contrary to that reported in the literature for zeolites, with the mesoporous acids fairly high selectivities are observed at high conversions, which may be explained by the presence of the large unidimensional mesopores which promote the reaction of xylose to furfural by allowing fast diffusion of furfural out of the catalyst once formed, thus avoiding extensive consecutive degradation reactions. The reaction conditions such as residence time, temperature, solvent, catalyst/xylose ratio were optimized. Catalyst deactivation was observed after long residence times possibly due to the interaction of reaction products with the acid sites leading to surface loading. On the hydrodynamic behaviour of liquid-liquid dispersions, new experimental data and modelling results of drop size distributions in agitated contactors were obtained and published.

SECTION 3

ACTIVITY PLAN 2005

Follows the activity plan for 2005 for the same areas and lines of study:

AREA 1 - ADVANCED MICRO- AND NANO-STRUCTURED MATERIALS FOR COMMUNICATIONS TECHNOLOGIES

Inorganic Multifunctional Materials and Organic-Inorganic Hybrids

Electroceramics

Magnetostructural Modulation of Strongly Correlated Electric Materials

Advanced Molecular and Supramolecular Materials

AREA 2 - ADVANCED MATERIALS FOR INDUSTRIAL APPLICATIONS

Reactive Ceramic Components for Process Control

Ceramic Composites and Ultra-Hard Coatings for Mechanical Applications

AREA 3 - CHEMISTRY AND TECHNOLOGY OF POLYMERIC AND LIGNOCELLULOSIC MATERIALS AND BIOMATERIALS

Macromolecular Materials and Lignocelullosics

Biomedical and Biomimetic Materials

Process Development and Optimisation

AREA 1 - Advanced Micro- and Nano-Structured Materials for Communications Technologies

INORGANIC MULTIFUNCTIONAL MATERIALS AND ORGANIC-INORGANIC HYBRIDS

New Microporous Materials. Work on microporous lanthanide silicates will continue. In particular the preparation of films and membranes of microporous materials (luminescent or not) will be pursued along two lines, the syntheses of: (i) of Eu-doped ETS-10 and umbite membranes; and (ii) new titanosilicate umbite membranes for the separation of H_2 . Microporous lanthanide silicates, AV-9 materials, will be evaluated as magnetic resonance contrast agents. Attempts to engineer magnetic centres into microporous materials resulted will focus on copper silicates. Applications of microporous titanosilicates and zirconosilicates for uptake of Hg^{2+} from aqueous solutions will be explored. The characterization of Y zeolites dealuminated by solid-state reaction with ammonium hexafluorosilicate will be studied.

Hypothetical uninodal zeolitic structures (containing one kind of tetrahedral sites) systematically enumerated using tiling theory have now been thoroughly characterised by computational chemistry methods and their feasibility evaluated taking into consideration relevant optimised structural parameters (e.g., unit cell dimensions and crystallographic symmetry), framework energies relative to α -quartz and volumes accessible to sorption. The main goal for 2005 is to sort out all the binodal and trinodal zeolitic frameworks (containing two and three kinds of tetrahedral sites, respectively) using the same systematic approach based on the above mentioned physical parameters. Furthermore, as the number of new bi- and trinodal topologies is comparatively much larger than that of the uninodal frameworks, structures will be described in terms of families by using a typical model-building approach (i.e., the presence of common Secondary Building Units, which characterise a given family of frameworks).

Mesoporous Materials. Work will continue to functionalize ordered mesoporous silicas with multidentate ligands and use these materials as supports for the heterogenisation of metal complexes with interesting catalytic or photophysical properties. For example, a recent literature method will be followed for the anchoring of bidentate pyrazolylpyridine ligands (L) onto MCM-41. This material will be exposed to lanthanide complexes of the type $Ln(NTA)_3$ ($Ln = Eu, Gd$; $NTA =$ naphthoyltrifluoroacetone) with the aim of preparing tethered adducts $Ln(NTA)_3 \cdot L$. The photoluminescence behaviour of the supported complexes will be studied and compared with model complexes bearing bidentate pyrazolylpyridine, bipyrimidine and phenanthroline ligands. MCM-41 derivatized with the pyrazolylpyridine ligand will also be examined as a support for CH_3ReO_3 .

Layered Materials. Upon calcination, the layered lanthanide silicates, $K_3[M_{1-a}Ln_aSi_3O_8(OH)_2]$ ($M = Y^{3+}, Tb^{3+}; Ln = Eu^{3+}, Er^{3+}, Tb^{3+}, \text{ and } Gd^{3+}$), named AV-22 materials, transform into a tunnel structure. This process and the photoluminescence properties of the resulting materials will be studied.

Work will continue on the intercalation of metallo-organic complexes with photofunctional or catalytic properties into layered double hydroxides (LDHs). Two main synthetic strategies are envisaged for this work. The first involves the incorporation of anionic complexes by ion-exchange reactions with LDH precursors containing either simple anions such as chloride or organic anions such as toluenesulfonate. Precursor materials with different M^{II}/M^{III} ratios (Zn^{II} , Mg^{II} , Al^{III} , Cr^{III}) will be prepared by co-precipitation of the component hydroxides under controlled pH conditions. The anionic metal complexes will include oxometal complexes of the type $[MO_2L_2]^{2-}$ ($M = Mo, W, Os$; $L =$ hydroxyl carboxylate ligand). We also intend to prepare some chiral complexes using, for example, quinic acid, or Schiff-base ligands derived from salicylaldehyde and chiral amines. Other complexes to be studied include anionic tetrakis(β -diketone) lanthanide derivatives and ruthenium cyanocomplexes $[Ru(CN)_x(L)]^m$. The second synthetic strategy involves the immobilization of metal complexes in LDHs containing transition-metal liganding moieties such as 2,2'-bipyridine-5,5'-dicarboxylate. The "solid ligands" will be prepared by ion-exchange, and the metal species introduced by a ligand exchange reaction or direct complexation. Selected materials will be tested as catalysts for the oxidation of organic compounds, including asymmetric oxidations in the case of chiral guests. The main reaction to be studied will be the epoxidation of olefins using molecular oxygen or hydroperoxides as the oxygen source. The results will direct further synthetic work, with the aim of producing truly heterogeneous catalysts that can be recycled without loss of activity or selectivity.

Nanostructured Materials. The growth of semiconductor nanocrystals at the surface of inorganic substrates will be investigated. Several experimental parameters will be investigated in order to propose a possible mechanism for the growth of such nanocrystals at the inorganic surfaces. In a first stage studies will be performed on the CdSe semiconductor due to the observation of strong quantum size effects. ZnO quantum dots will be prepared using colloidal synthetic approaches. The ZnO QD's will be doped with distinct lanthanide ions and their photoluminescence properties will be then investigated. Polymer nanocomposites consisting on nanostructured fillers dispersed homogeneously with polymer matrices will be prepared and investigated.

Novel Pigments. Nanocomposites containing inorganic particles (e.g. TiO_2) and cellulosic fibres will be investigated. Morphosynthetic studies on $BiVO_4$ pigment particles will be performed, considering the influence of experimental parameters such as chelate agents and foreign metal species.

Polyoxometalates. The synthesis and study of new hybrid compounds with polyoxometalates and organic moieties will be continued, namely aminoacids (histidine, tryptophan) and aromatic imines or amines. A new synthetic method is being developed in order to be applied to the preparation of some of the compounds already obtained by more complex procedures. The compounds will be studied for properties like photochromism or non-linear optics properties. Oxidative catalysis studies will continue. It is also planned to proceed further with the development of electrodes incorporating Keggin and sandwich type polyoxometalates and study their possible applications in analysis and electrocatalysis.

Novel Luminescent Systems. The aims of research in this topic are the preparation of novel lanthanide luminescent systems at the molecular or supramolecular level together with systems supported in a nanosized material.

We will attempt to prepare full-colour phosphors based on a dense system isostructural with $\text{Na}_3\text{YSi}_3\text{O}_9$.

Research will be continued on the synthesis of photoactive lanthanide complexes with aromatic ambidentate ligands, exploring the possibility of formation of multidimensional coordination compounds. The luminescence and structural properties will be thoroughly studied. The possibility of the ligands to function as remote light-harvesting units, acting as an antenna for collecting light and transferring the energy to the lanthanide, will be particularly investigated.

Preparation and structural characterization of ligands based on lacunary polyoxomolybdates or polyoxotungstates (eg. Keggin-type lacunary tungstophosphate and tungstoborate anions) will be continued. We will investigate the preparation and structural characterization of new organic/inorganic hybrid coordination networks using lanthanopolyoxometalates and aromatic ambidentate ligands. The application of the compounds in the preparation of polyoxometalate based materials will be explored, namely by the preparation of mono or multilayered nanostructured films and polyoxometalate-anion-pillared layered double hydroxides.

Crystal Engineering of Organic-Inorganic Hybrids. The synthesis of novel hybrid materials (containing both transition metal centres and/or lanthanide cations) will continue to be focused on the simultaneous use of flexible organic ligands combining several functional groups with a multitude of coordinating capabilities [such as N-(phosphonomethyl)iminodiacetic acid and etidronic acid], along with bridging organic molecules traditionally used in this field of Crystal Engineering (e.g., pyrazine, piperazine and 4,4'-bipyridine). In this context, the centrosymmetric $[\text{V}_2\text{O}_2(\text{pmida})_2]^{4+}$ units are expected to play a decisive role as Secondary Building Units to construct novel multi-dimensional frameworks. In particular, the effect of the use of pyrazine (instead of 4,4'-bipyridine) in the final topology of the frameworks belonging to the previously reported $[\text{MVO}(\text{pmida})(4,4'\text{-bpy})(\text{H}_2\text{O})_2]\cdot(4,4'\text{-bpy})_{0.5}$ (where $\text{M} = \text{Cd}^{2+}$ or Co^{2+}) system will be studied. Similarly to that described for the reactions between N-(phosphonomethyl)iminodiacetic acid and V^{4+} and Ge^{4+} , we are expecting etidronic acid to form interesting SBUs with these metal centres. These building blocks will be comprehensively characterised and used along with transition metal and/or lanthanide cations in the construction of novel frameworks which are expected to have functionality (e.g., magnetism and/or luminescence).

Organic-Inorganic Hybrids Lacking Activating Centers. Work on the discussion of the nature of the emission of white-light photoluminescence sol-gel derived amine-functionalized hybrids lacking metal activator will continue. The focus will be on the discussion of the energy transfer mechanisms between the two emission components and on EPR studies. New class II poly(ethylene)/siloxane composites, classed as *amidosil*, where the organic and inorganic counterparts are covalently bonded through amido bridges will be synthesized and characterized. The cross-linkages can exist in both sides of the polymer chain or only in one of the terminal sides. The structure, the luminescent properties and the emission quantum yield of mono/di-

amidosils with different polymer chains will be studied. The effect of the cross-linkages at both ends or at one side of the polymer chain and the role of the self-organization on the PL features (both in steady state and time-resolved modes) is under progress. The theoretical model behind the emission processes, considering that the excited carriers can move within localized states accordingly to the extended multiple trap model, will be improved and tested for several families of amine-functionalized hybrids.

New Hybrid Materials. In order to investigate the ability of different hybrid materials to efficiently incorporate lanthanide ions, protecting them from non-radiative channels, thus enhancing the respective PL features the following organic-inorganic hybrids will be synthesised and investigated: Di-ureasils doped with $\text{Er}(\text{CF}_3\text{SO}_3)_3$ are multi-wavelength emitters from the visible to the infrared spectral region due to the overlap of the blue-green spectral typical luminescence of the hybrid host with the Er^{3+} emission around 1530 nm. The observation of efficient RT from the lanthanide ions show that the di-ureasils can act as a good cage-type host to incorporate Er^{3+} , demonstrating capacity of well shielded from interaction with non-radiative paths. The di-ureasils modified by $\text{Eu}(\text{NTA})_3\cdot\text{bpy}$ ($\text{NTA}=1-(2\text{-naphthoyl})-3,3,3\text{-trifluoroacetate}$, $\text{bpy}=2,2'\text{-bipyridine}$) complex display the typical Eu^{3+} red emission, due to an efficient intra-energy transfer between the hybrid host emitting centres and the complex. Although the structural and emission features do not depend on the synthesis route (conventional sol-gel method and acetic acid derived solvolysis), different emission quantum yields values were obtained for the hybrids prepared through the solvolysis and by classical sol-gel. The interpretation of this result is still under discussion.

The magnetic characterization of bio-inspired iron-doped di-ureasils will continue. We intend to explore the possibility of self-organization (via hydrogen bonds between adjacent urea groups) in the matrix performing the synthesis under a magnetic field. Furthermore, the synthesis of di-ureasils incorporating other iron-oxide phases with high magnetic moments will be attempted. Complementary characterization techniques such as transmission electron microscopy and Mossbauer spectroscopy will be planned.

Planar waveguides with low losses in the infrared (from 0.6-1.1 dB/cm) will be prepared with sol-gel derived poly(oxyethylene)/siloxane di-ureasils doped with zirconium (IV) n-propoxide and methacryloxypropyltrimethoxysilane. Preliminary PL results indicate an effective interaction between the zirconium particles and the siliceous nanodomains.

C60 Phase Transitions Under High-Pressure. The structures of polymeric 3D C60 phases will be determined definitely. New in-situ diffraction experiments at the European Synchrotron Radiation Facility (up-grade of the high-pressure beam-line had cancelled experiments planned to 2004) are scheduled in order to study the structural properties of a ferromagnetic phase ($T_c\sim 500\text{K}$) found during the amorphisation process of polymeric C60.

Development of Spectroscopic Techniques. A new solid-state NMR method will be introduced, using multiplex phase cycling to improve the efficiency of 3Q and 5Q MQMAS NMR.

The potential of Surface-Enhanced Raman Scattering (SERS) for studying the interaction of metal nanocrystals with molecular adsorbates has been explored, in particular for the investigation of adsorption modes and orientation of molecules on the surfaces. Research will be continued on the use of SERS as a tool for trace detection. SERS will also be used to characterise systems with potential interest in heterogeneous catalysis. Techniques of infrared spectroscopy applied to surface studies will also be explored.

ELECTROCERAMICS

Microwave Ceramic Dielectrics. We shall concentrate on the elucidation of the relations between microwave dielectric properties and composition, structure and microstructure of perovskite ceramics based on $\text{La}(\text{Mg}_{1/2}\text{Ti}_{1/2})\text{O}_3$ (LMT). Attention will be paid to the processing of these ceramics in order to get homogeneous, single-phase and dense material as a basis for substitutions at A- and B-sites of the perovskite structure. Mechanisms governing dielectric loss and τ_f are the main focus of the project. Specific objectives for LMT-based systems, as a direct follow of previous work, aims to increase Q to interesting values for resonators and lower sintering temperature to enlarge the scope of application to LTCC and NPO devices. This can be achieved through doping but also by new solid solutions where La is replaced by Na or Bi. Ferroelectric-relaxor transition will also be studied for a particular area of solid solutions compositions.

Measurements of dielectric characteristics at radio and microwave frequencies will be performed for the LMT based compositions with different substitutions. For the assessment of the major contributing mechanisms it is intended to continue to use far infrared spectroscopy. Structure analysis will be used involving techniques such as XRD Rietveld refinements and TEM. The study of the effect of sintering atmosphere will also be useful to understand the role of certain defects in the dielectric properties.

Ferroelectric Ceramics. Microstructural design of PZT ceramics will continue through the investigation of the type of liquid phase and thermal schedules favouring a high grain growth rate in these ceramics.

PZT single crystals will be prepared by the method of high temperature flux growth and used as seeds in the PZT ceramics to favour the occurrence of abnormal grain growth during sintering.

The dependence of core shell formation on the annealing treatments of PFW – PT ceramics will be performed. By controlling the cooling ratio after sintering or by performing additional annealing treatments the microstructure and dielectric properties of PFW-PT ceramics will be evaluated and the mechanisms of the core-shell structure formation and collapse in this system will be proposed.

Ferroelectric Fibres, Single Crystals and Films. Acrylic acid showed to be the best candidate to create long linear polymeric chains in the fibres gel precursors and to improve the macroscopic properties of PZT fibres. In order to clarify the role of the

acids on the molecular structural development of the gel Gas Chromatography and NMR studies will be conducted and complemented with the microstructure evolution.

Preparation and characterisation of SBT(SBN) thin films using seeding and doping approaches - SBT(SBN) seeds produced by the chemicals methods already reported will be used to improve the synthesis of the corresponding thin films by a solgel procedure, aiming to lower its crystallization temperature and to improve their ferroelectric properties. Characterization of the unseeded and seeded thin films (XRD, SEM) will be combined with annealing procedures to access the effects of the seeds on the crystallization of the perovskite phase and on the thin film microstructure. The dielectric and ferroelectric properties of the so obtained thin films will be investigated. The existing correlations between the final properties and the thin film processing conditions will be evaluated so as to detail the role of the seeds. A different approach, i.e. doping, will also attempt to improve the properties of the thin films. Suitable ions will be selected for this study and their effect on the ferroelectric properties of the films (remanent polarization, coercive field, fatigue endurance,...) will be investigated.

Growth of SBN single crystals by high temperature self-flux solution method - the growth of $\text{SrBi}_2\text{Nb}_2\text{O}_9$ (SBN) single crystals will be investigated. The high temperature self-flux solution method used with success for growing SBT single crystals will be improved for this new composition: several experimental variables (flux amount and composition, cooling profile) will be studied aiming at identifying optimum conditions for obtaining large sized single crystals with high quality. The electrical properties of the obtained crystals will be characterized at several frequencies and temperatures.

In order to decrease the processing temperature and to increase the orientation of the films the effect of the seed layer and its thickness on the texture degree and final electrical properties of BST thin films will be studied. The dielectric constant and loss tangent, leakage current and remnant polarization will be evaluated. The correlation between the orientation and the dielectric properties will be discussed.

Incipient Ferroelectrics. The work will continue along three main lines: (i) continuation of the structural characterisation of Mn doped ST ceramics, namely at low temperature by Raman, RXD and TEM, in order to clarify the differences in the phase transition temperature induced by the different lattice site occupancy; the dielectric properties will be measured as a function of temperature and frequency, in a wide frequency range, in order to establish the relations between the structure and the dielectric response of ST-Mn doped samples; (ii) continuation of the sintering and dielectric studies of the effect of nonstoichiometry on the incipient ferroelectric behaviour of ST, (iii) continuation of the studies on the doping effect on SrTiO_3 thin films deposited on different substrates. The dopants under study will be Bi, Mg and Zn. The films will be characterised from the structural, microstructural and dielectric point of view.

Nanoscale Properties of Ferroelectrics. The investigations by Scanning Force Microscopy of the local properties of ferroelectric thin films, single crystals and ceramics will be continued. We plan to study in detail the local properties under a high electric field. The formation of the nanoscale domains of the size approaching to the size of the SFM tip will be attempted using the effect of universe poling observed in 2004. The investigations will be extended to a wide range of the

compositions and thicknesses of the PZT films of different textures. Piezoelectric ceramics of the commercial compositions, piezoelectric composites and multilayer actuators will be investigated at the nanoscale with the aim of improving their electromechanical performance. We plan to extend our activity to antiferroelectric thin films where it could be possible to locally induce a antiferroelectric-ferroelectric phase transition and to investigate the nature of the antiferroelectric state at the nanoscale level. We also plan a new activity which has to deal with the nanolithography and nanopatterning with SFM.

We will continue the studies using Scanning Force Microscopy of ferroelectric relaxors including new compositions and new measurement techniques in order to obtain new information on the nature of the polarization state in relaxors. Pure PMN and PLZT that should freeze at low temperatures will be investigated using Peltier cold stage. New objects of the investigations will include PMT, SBT:Ce and PFW. The comparison with the results of neutron scattering will be done in order to confirm the mean size and distribution of polar clusters with obtained by independent technique. Finally, a new measurement procedure using synchrotron radiation will be attempted.

The study of the nature of giant piezoelectric response in PZN-PT single crystals will be extended to poled single crystals. Comparing of the domain structures of poled and poled crystals

The nanoscale study of ferroelectric fatigue in ferroelectric ceramics and thin films study will concentrate on the nature of defects responsible for fatigue. The nanoscale measurements will be complemented with thermally stimulated currents, conductivity, and dielectric impedance spectroscopy that all will be used to achieve this goal. Finally, a new physical phenomena: asymmetric strain hysteresis as a result of fatigue will be investigated.

MAGNETOSTRUCTURAL MODULATION OF STRONGLY CORRELATED ELECTRIC MATERIALS

Colossal Magnetoresistive Materials. a) Continue the work on thin film preparation, structural (X-ray), magnetic and electrical properties. Study of magnetic and transport properties of CMR manganites and other perovskite derivatives of series R-Ca/Sr-MnO₃ (R=La, Pr, Eu, Er, Gd) and Mn substitutions (Cr, Cd); b) Assembly of a RF sputtering deposition system for oxide thin films; c) Studies using implanted radioactive isotopes at ISOLDE-CERN; d) Theoretical study of phase transitions and application of non-extensive statistics to manganites; e) Magnetocaloric effects.

High Temperature Superconductors. Physical characterization studies of oxide superconductor materials prepared by LFZ: magnetic and electrical measurements.

ADVANCED MOLECULAR AND SUPRAMOLECULAR MATERIALS

Hydrogen Bonds. The role of hydrogen bonds in the structure and properties of molecular and supramolecular materials will be accessed for a range of organic, inorganic and hybrid systems, using *ab initio* calculations and experimental techniques

(vibrational spectroscopy, NMR spectroscopy, inelastic neutron scattering and X-ray crystallography). Particular attention will be given to the importance of this kind of interaction in materials of the so-called “class I” (in which the molecular unities are bound through non-covalent interactions). Properties related with the hydration of crystalline samples (pseudo-polymorphism) and energy transfer in hybrid luminescent materials will be addressed during 2005.

CYCLODEXTRINS. NMR studies of the cyclodextrin/decanoic (or hexanoic) acid systems in the presence F^- will be used to determine the conformation of the guest molecule, equilibrium constants and aggregation properties of decanoic/hexanoic acid included in β -cyclodextrin. The geometry of inclusion of decanoic acid in alfa- and trimethyl-cyclodextrin will also be studied by NMR.

Studies on cyclodextrins (CDs) as second sphere ligands for metal complexes will continue. Complexes of the type $(FcNN)M(CO)_4$ ($M = Mo, Cr$) and $MoO_2Cl_2(FcNN)$ bearing the ligand N,N' -bis(ferrocenylmethylene)ethylenediamine (FcNN) will be prepared and immobilized in the modified cyclodextrin heptakis-2,3,6-tri-*O*-methyl- β -CD (TRIMEB). It will be assessed whether the behaviour of the complex $MoO_2Cl_2(FcNN)$ as a catalyst for the liquid phase epoxidation of olefins can be modified beneficially by interaction with TRIMEB. For example, encapsulation may enhance solubility, stabilize the catalyst and also facilitate subsequent recycling. In another study, inclusion compounds comprising molybdenocene dichloride (Cp_2MoCl_2) and the cyclodextrins β -CD, TRIMEB and 2-hydroxypropyl- β -CD will be prepared and characterized using the techniques listed above. The adducts will be screened for their potential antiproliferative and cytotoxic activity, in both human cancer and healthy cell lines.

Oxomolybdenum Catalysts. We have recently published a series of papers focusing on complexes of the type $[MoO_2X_2L]$, which are versatile, excellent catalyst precursors for olefin epoxidation in the presence of *tert*-butyl hydroperoxide (TBHP). Important properties, such as the solubility of the complex and the Lewis acidity of the metal centre, can be fine-tuned by variation of X and L. Dioxomolybdenum(VI) complexes with polypyridyl ligands such as bipyridine and bipyrimidine are rather sluggish catalysts of low general activity. Better results have been obtained using substituted 1,4- R_2 -diazabutadienes as supporting ligands. Future work will continue to examine the effects of ligands in dioxomolybdenum(VI) complexes. For example, bidentate imines are particularly attractive since they provide considerable scope for the variation of steric and electronic properties. The catalytic properties of the new family of complexes will be explored.

Outstanding results have also been achieved with the novel cationic complex $[MoO_2Cl(Bn_3Me_3-tame)]BF_4$ [$Bn_3Me_3-tame = N,N',N''$ -tribenzyl-1,1,1-tris(methylaminomethyl)ethane] as catalyst for cyclooctene epoxidation using TBHP as the mono-oxygen source under mild reaction conditions. Therefore, a series of ionic dioxomolybdenum(VI) complexes of general formula $[LMoO_2X][Y]$, containing tridentate nitrogen ligands, will be prepared and tested as catalysts for the production of high added value olefins, such as limonene oxide.

The industrially applied oxidation agent in the ARCO/Halcon epoxidation process is TBHP. It is therefore of significant

interest to gain a deeper insight into the mechanism of the epoxidation with the above catalytic systems. The debate has not been settled to date, despite the fact that a lot of information has been derived from NMR and catalytic reactivity patterns, and several theoretical and mechanistic studies have been presented. Further studies on the mechanism of the epoxidation reaction with novel catalysts will be carried out with the aid of several spectroscopic techniques in combination with GC/MS.

Novel Transition Metal Complexes. The synthesis and characterisation of novel polypyridyl transition metal complexes and supramolecular mixed metal complexes of the type $[(M(9[ane]S_3)(4,4-bpy)_2)(M'(bpy))_2]^{8+}$ will be continued. Characterization via ES-MS, capillary electrophoresis and DOSY NMR will be carried out. The interaction of selected complexes with metal transport proteins will be probed using NMR experiments such as tr-NOESY, DOSY and STD.

Complexes for DNA probes and Photo and Electrochemical Applications. Designed compounds capable to display DNA-specific binding characteristics, together with reduced toxicity (for DNA molecular recognition), with transition metals and polypyridyl and/or thioether ligands will continue to be synthesised and characterised by the usual techniques. Bridging ligands will continue to be tested to isolate dinuclear systems to evaluate their photo and electrochemical properties. MS advanced techniques will be used in the identification and fragmentation patterns of new synthesised binuclear complexes.

Macrocycles. Experimental (X-ray single crystal diffraction analysis) and theoretical studies in a wide range of inorganic and organometallic systems, with a particular focus for complexes containing macrocyclic ligands, namely the study of the interaction host-guest in supramolecular systems involving macrocrocycles and organic substrates or metal ions. Theoretical methods will include molecular mechanics, molecular dynamics and quantum mechanics calculations. Other studies in the domain organometallic crystal engineering, analysis of intermolecular interactions and the understanding of molecular recognition mechanisms (for example chiral resolution of racemate mixtures by chiral silicon phases will be carried out.

AREA 2 – Advanced Materials for Industrial Applications

REACTIVE CERAMIC COMPONENTS FOR PROCESS CONTROL

Materials For High-Temperature Electrochemical Applications. Work on solid electrolytes will be extended to re-examine the limitations of alternative materials, mainly those of lower cost. Some effects to be addressed in studies of $\text{La}_{10}\text{Si}_6\text{O}_{27}$ -based apatites will be the reducibility of silica to volatile SiO and the onset of electronic contributions, mainly for cases when aliovalent additives (e.g. SrO) or additives with mixed valency (e.g. Fe oxides) are used to enhance the conductivity or to improve the sinterability. Detailed studies of transport properties will also be used to re-examine the defect chemistry of some materials. We expect to obtain confirmation that interstitial oxygen ions are the predominant carrier in this type of materials, and to attain a better understanding of mechanisms of ionic conduction, including the role of dopant cations such as Sr^{2+} . The effects of Fe^{n+} will be examined in detail.

Further work on $\text{La}_2\text{Mo}_2\text{O}_9$ -based materials will be performed aiming to extend the stability limits and electrolytic domains under reducing conditions, to understand and prevent a \rightarrow b transformation, and to lower the thermal expansion.

Impedance spectroscopy will be used for electrical characterization of potential cationic conductors (e.g. $\text{K}_x\text{M}_{(1+x)/3}\text{Sb}_{(2-x)/3}\text{O}_2$, with $\text{M} = \text{Ni}^{2+}, \text{Mg}^{2+}, \text{Co}^{2+}$), including the role of internal interfaces or other microstructural features, and attempts to interpret their interaction with H_2O -containing atmospheres. Structural refinements will be performed to attain a better understanding of cationic transport in these layered structure-types.

Mixed conductors are also being assessed as components of electrodes for reducing conditions such as anodes of solid oxide fuel cells. This work encompasses different structure types, such as fluorites ($\text{Ce}(\text{Pr},\text{M})\text{O}_{2-\delta}$ with $\text{M} = \text{Zr}, \text{Gd}, \text{TbZrO}_{4-\delta}$), zircon-type ($\text{Ce}_{0.8}\text{Ca}_{0.2}\text{VO}_{4+\delta}$), pyrochlores ($\text{Gd}_{1.86}\text{Ca}_{0.14}\text{Ti}_2\text{O}_{7-\delta}$) and perovskites ($\text{La}_{1-x}\text{Sr}_x\text{Al}_{1-y-z}\text{Mg}_y\text{Fe}_z\text{O}_{3-\delta}$). $\text{Ce}(\text{Pr},\text{M})\text{O}_{2-\delta}$ with $\text{M} = \text{Zr}, \text{Gd}, \text{YBaCo}_4\text{O}_{7+\delta}$). Novel mixed conductors (e.g. $\text{Sr}_{0.7}\text{Ce}_{0.3}\text{Mn}_{1-x}\text{Al}_x\text{O}_{3-\delta}$) are also being considered as alternative oxygen electrodes for high temperature electrochemical applications.

Glass-ceramic sealants for SOFC-based systems are being studied, based on SiO_2 - Al_2O_3 - CaO - BaO and SiO_2 - MgO - BaO systems. This work includes the compatibility with zirconia, ceria or lanthanum gallate-based electrolytes for solid state electrochemical applications.

Mixed Conducting Materials For Oxygen Separation or Partial Oxidation of Hydrocarbons. Since most mixed conductors tend to favour complete oxidation in membrane reactors, we will re-examine the electrocatalytic activity of a variety of mixed conductors containing elements such as Fe-, Co-, Ni-, Ce and V; this includes a wide range of materials, such as perovskites ($\text{SrFe}_{0.7}\text{Al}_{0.3}\text{O}_{3-\delta}$, $\text{La}_{0.3}\text{Sr}_{0.7}\text{Co}_{0.8}\text{Ga}_{0.2}\text{O}_{3-\delta}$), dual-phase composite such as $(\text{SrCo})_{0.5}(\text{Sr}_2\text{Fe}_3)_{0.5}\text{O}_{4.75\pm\delta}$, La_2NiO_4 -based materials, zircon-type $\text{CeVO}_{4+\delta}$, $\text{CeNbO}_{4+\delta}$, etc. Mixed conductors will be tested as disk-shaped membrane reactors, with and without catalysts, by assessing the conversion of methane+oxygen+inert mixtures in contact with different catalysts, and by pulsing

CH₄+inert over mixed conductor powders. On will attempt to establish correlations between the kinetics of methane oxidation and bonding energy between oxygen and transition metal cations, oxygen desorption and oxygen ionic transport, in order to find criteria for selecting the most promising materials. So far, our best results in terms of methane conversion and CO selectivity have been obtained for SrFe_{0.7}Al_{0.3}O_{3-δ}. The transport properties of this material will thus studied in detail, and a Mössbauer spectroscopy will be used to assess the role of surface states of iron and oxygen ions in the catalyst. Other studies include the roles of phase transformations, partial decomposition, oxygen non-stoichiometry and/or defect ordering under typical reducing atmospheres containing CH₄, H₂ and/or CO. Changes in composition (e.g. SrFe_{1-y}Al_yO_{3-δ} and Sr_{1-x}Ca_xFe_{1-y}Al_yO_{3-δ} ceramics) will be introduced to optimise the relevant properties and catalytic performance.

Further studies on mixed conducting materials for oxygen separation or selective oxidation will be focused on better understanding of the defect chemistry and transport properties of highly stoichiometric materials, namely: perovskite materials based on (La,Sr)FeO₃, (La,Sr)CoO₃, Sr(Ti,Fe)O₃, Ca(Ti,Fe)O₃ and LaGaO_{3-δ}; and hiperstoichiometric La₂NiO_{4+d}-based materials.

p(O₂)-T-δ diagrams will be evaluated by coulometric titration, and combined with transport property measurements obtained from the dependence of Seebeck coefficient and total electrical conductivity to derive defect chemistry diagrams. Atomistic modelling will be used to re-examine or interpret those data.

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We will also extend the work on mixed conducting composites comprising one electronic conductor, such as La_{0.1-x}Sr_xFe_{1-y}Co_yO_{3-δ} (LSFC), La_{1-x}Sr_xMnO_{3-δ} (LSM) or La₂Ni_{1-x}Cu_xO_{4+δ} (LNC), and one ionic conductor based on zirconia (YSZ), ceria (CGO) or lanthanum gallate (LSGM). Phase interaction between different combinations (e.g. LSM+YSZ, LSCF+LSGM, LSCF+CGO), will be used to establish guidelines to optimise processing conditions and materials design.

Catalysts For Selective Oxidation of Hydrocarbons. The prevailing mechanism of total methane oxidation observed for most membrane materials makes it necessary to incorporate reforming catalysts in the membrane reactors for natural gas conversion to synthesis gas. Attempts to obtain catalysts for selective oxidation of hydrocarbons include the preparation and characterization of nanocrystalline Ce_{0.45}Zr_{0.45}La_{0.10}O_{2-δ}, a variety of mixed conductors and other potential catalysts containing two redox pairs (e.g. tetragonal CeNbO_{4+δ}).

Microstructural Effects. We will extend the analysis of grain boundary effects on solid electrolytes or mixed conductors, with an emphasis on the effects of sintering additives on microstructures and the corresponding effects on transport properties; this includes ceria- or zirconia-based electrolytes and protonic or cationic conductors. Novel processing methods are also being studied to obtain ionic or mixed conductors with enhanced transport and/or electrocatalytic properties. Ongoing studies are cellulose-precursor synthesis of electrocatalytically active components of SOFCs and mixed-conducting membrane reactors and preparation of materials of submicrometric materials (CaFe_{0.2}Ti_{0.8}O_{3-d}) by mechanical activation.

Materials For Other Electrochemical Technologies. Alternative electrochemical methods are also being considered for other technologies, including the electrowining of metals, alternative low CO₂ steelmaking technologies, and other environmentally friend processes. Ceramic materials are being studied as potential electrodes and electrocatalytics for such electrochemical methods.

CERAMIC COMPOSITES AND ULTRA-HARD COATINGS FOR MECHANICAL APPLICATIONS

Diamond Coatings. Nanocrystalline diamond (NCD) films on silicon nitride ceramics are being developed for tribological applications. This project aims at the development of NCD coatings for unlubricated and boundary lubricated tribosystems, such as machine elements and metalworking processes requiring low friction and high wear resistance; and cutting tools of highly abrasive materials. This includes very smooth NCD tools, which may excel PCD and CVD diamond tools in machining of abrasive materials like hardmetal, MMCs and CFRCs.

A combination of Si₃N₄-bioglass composites with an ultra-hard biocompatible NCD film will be developed aiming at bio applications such as joint implants, anticipating extended lifetime and resistance to sterilisation procedures.

Wear mechanisms on hard and ultra-hard materials (Si₃N₄) with CVD diamond tools will be studied for the optimization of the wear resistance of new CVD diamond tools produced by CVD thin film (ap. 10mm) direct coating. The grain size of the direct coatings varies in the range of nanodiamond (<100 nm) to polycrystalline diamond (2-10 mm). These cutting materials have already been tested in turning of hard metal and work will continue on graphite and ceramic materials machining. Turning tests are assessed by real-time forces acquisition and subsequent evaluation techniques: tip wear measurement by SEM and AFM, analysis of the film residual stresses by Raman spectroscopy, evolution of the cutting edge surface roughness and evaluation of surface finishing quality of the workpiece by profilometry.

The tribological properties of CVD diamond coatings on Si₃N₄ will also be studied. Self-mated CVD diamond tribological systems for machine elements (e.g. seals, valves, bearings) require low friction and high wear resistance. Reciprocating sliding ball-on-flat wear tests will be conducted with or without lubrication, at variable normal load, involving CVD diamond coatings, ranging from nanocrystalline to microcrystalline. Friction and wear results together with characterisation with several techniques (SEM, AFM, micro-Raman) will lead to comprehension of the friction and wear mechanisms.

Other Hard and Ultra-Hard Materials. Hardmetal grades with sub- to nano-metric grain size with superior erosive wear resistance will be studied as innovative solutions for applications with intensive erosion, such as oil and natural gas extraction, chemical and pharmaceutical plants. New hard-metal compositions will be developed from sub-micrometric (0.7-0.8 μm), ultrafine (0.1-0.6 μm) and nanopowders (< 100 nm) with reduced amounts of binder phase (1-6% in weight). The densification of the new grades will imply pressure-assisted sintering and optimisation of conventional sintering. Improved microstructures are expected to yield adequate mechanical properties (hardness, fracture toughness) and much improved

erosion wear resistance relatively to the standard micrometric grades. Component prototypes will be produced for in-service tests in industry.

Ceramic-metal composites based on TiC and high temperature intermetallics will be obtained by infiltration. These studies include the infiltration kinetics and the load bearing capability of these composites at high temperature. High-temperature bending strength and the creep resistance of different composites will be evaluated and related to the microstructure and phase composition. Load bearing of the different phases and their contribution to the overall high temperature mechanical response of TiC containing composites will be assessed. Different intermetallics, Ni₃Al, NiAl, Fe₂₈Al and Fe₄₀Al will be studied.

Hard coatings on steels for aluminium injection will also be studied.

Colloidal Processing. Methods will be developed to process advanced ceramic and glass-ceramic materials by colloidal shaping techniques using preferentially aqueous media. Non-aqueous media will be used for multicomponent systems that might exhibit some incompatibility in water. New direct shaping methodologies are under development, which will enable de consolidation of large and homogeneous ceramic parts. Such systems are also expected to perform well in rapid manufacturing by, for example, ink-jet printing.

Macroporous ceramic bodies will be prepared with tailored porous microstructures for bone-ingrowth in biomedical applications. Mechanical, *in vitro* and *in vivo* characterization of the macroporous materials will be carried out in collaboration with other specialized research centres and hospitals.

Other Processing Methods. New glass and glass-ceramic compositions will be developed for different applications (structural, optical, biomedicine, electronics, etc.). This includes studies of the nucleation and crystallization processes and the properties of the resulting glass-ceramics.

Suitable methods will also be developed for recycling industrial wastes and by-products or incorporation in new added-value materials. The current research works will be extended to other types of residues in order to find suitable solutions to clean the environment, preserve natural and non-renewable resources, save energy, while improving some of the actually existing products or developing new ones with new functionalities.

AREA 3 – Chemistry and Technology of Polymeric and Lignocellulosic Materials and Biomaterials

MACROMOLECULAR MATERIALS AND LIGNOCELLULOSICS

Lignocellulosics. Investigations aiming to improve the retention of polysaccharides during *Eucalyptus globulus* kraft pulping will be pursued. The addition of borates and/or amines, together with modified profiles of active alkali and/or sulphidity will be investigated and optimized. The study of the conditions and factors determining the precipitation of xylans on the surface of fibres at the last stages of the kraft pulping will be pursued. Model systems including pure cellulose, xylans and lignins will be used in such studies. The development of mathematical models describing the selective precipitation of xylans and lignins will be initiated.

The development and characterization of new composite materials based on modified cellulose fibre with aliphatic moieties will be pursued. New cellulose fibre grafting agents, namely aliphatic polyethers and aliphatic hydroxyacids will be investigated and the modified fibres characterized. The thermal properties of modified fibres and thermoplastic composites will be assessed by DSC and DMA.

The synthesis of new cellulose fibre hybrids with CaCO_3 , ZnS or ZnO nanoparticles will be attempted. In parallel, the synthesis of cellulose/ TiO_2 hybrids, now using a recently proposed sol-gel approach, will be investigated. The hybrids will be characterized and, for selected materials, their papermaking properties will be assessed. Preliminary studies on the using of such hybrids or nanoparticles in composites will be initiated.

Research activities dealing with fractionation, characterization and new applications of cork components will be initiated within the scope of a European project. Cork and cork industry by-products (“black condensates” and “cooking waters”) will be sequentially extracted with solvents of different polarity. Cork will be submitted to alkaline hydrolysis aiming to depolymerize and extract suberin. The development, characterization and application of new thermoactive materials including cellulose fibres or cork will be pursued.

The new silica-cellulose hybrid materials will be tested for fire resistance and acoustic isolation. The similar sol-gel process will be applied for the modification of the paper surface.

The work on polyoxometalate (POM) catalysis in oxygen delignification using combinatory (alteration treatment of unbleached kraft pulp) approach with laccase in a multi-stage system for the oxygen bleaching of pulp will continue.

The studies on ESI-MS application for the structural characterization of lignin will continue.

A project on the improvement of the yield and the mechanical strength of acid sulfite pulp will continue. At the second step of this investigation the wood-to-pulping liquor ratio will be varied in order to estimate the effect of changes made in pulping on the pulp yield, mechanical strength properties and the bleachability. The conditions of chips pre-steaming the impregnation will be optimized.

The project on the improvement of sulfite pulp brightness in P(O)-P bleaching will continue. The post-treatment of bleached pulp with ozone and sodium hypochlorite will be tested aiming to increase the final pulp brightness.

The study on the hemicelluloses effect on the hornification of eucalypt bleached kraft pulp will initiate this year.

The investigation on the contribution of different kraft pulp components on its kappa number and brightness will start this year.

Other Polymer Systems and Materials. Work on the preparation of polymer based NC via living polymerisation mechanisms such as RAFT and ATRP in mini-emulsion will proceed. The collaboration with Carnegie Mellon Univ. will go on exploiting the application of ATRP to other systems. As regards the RAFT mechanism, the synthesis of thioesters and polymerisation studies will be carried out.

A MSc thesis will start on the preparation of polymer based NC and the study of their mechanical performance. The studies of polymer@SiO₂ nanoparticle interfaces will bring further control on NC preparation. The results obtained will contribute to the PhD programme on SiO₂/exopolysaccharide NC. In connection with this work a series of rheology studies will be carried out to assess the effect of SiO₂ nanoparticles. The preparation of NC based on conducting polymers will continue.

The work on amorphous and semi-crystalline polymer materials creep will continue and extended to the much more difficult stress relaxation processes. The development and experimental validation of accurate and workable, predictive, analytical (rather than simulative) non-linear temperature-dependent dynamic molecular models, for any levels of stress/strain, will be our most important objective.

A comprehensive Laboratory of Thermal Analyses will be set-up in 2005, to be fully equipped with power compensation differential calorimetry (DSC), simultaneous calorimetry and thermogravimetry (DSC/TG) and dynamic mechanical thermal analysis (DMTA), in combination with other associated facilities (at the IST, Lisbon) for thermo-stimulated polarization/depolarisation (TSDC), dielectrical thermal analysis (DETA) and other melt and solid rheometry. These facilities will be invaluable in the further development and evaluation of our recent cooperative segmental theory of molecular dynamics (CSTMD). These facilities will be used in an integrated way to probe extremely varied materials' structures within very wide ranges of temperature and time scales (from $< 10^{-4}$ to at least 10^8 Hz). The development of a sound and workable equilibrium and non-equilibrium thermodynamic theory of glasses will also be one long-term objective, along with the major one aiming at a general materials dynamics theory.

BIOMEDICAL AND BIOMIMETIC MATERIALS

Biological, Structural and Identification FTIR, NMR and Other Studies. New synthesized Cr(V) compounds will be tested in in vivo studies with mice, submitted to severe or chronic intoxication experiments, in order to localise degenerative damages in animal target organs and quantify the amounts of this element in those organs. Histology, histochemistry and ultra-structural techniques will be used to reach the purposed objectives. The results were then treated with occupational aims. Toxic effects of transition metal compounds are important in male fertility, concerning their relationship with industrial

processes. Comparison of traditional diagnostic techniques (biopsies) with faster, reliable and cheaper ones (flow cytometry and fluorescence microscopy) will be done intending the quantification of “dose-effect” relationships, again with occupational aims.

Within novel techniques, tools, and paradigms of imagiology for diagnosis, a wide range of techniques such as multispectral imaging, life-time imaging, near infrared imaging, polarization imaging and Z-scan imaging will be tested in animal cell cultures and tissues, as well as from patients, in order to create a database of high quality images for the diagnosis of some pathologies.

NMR relaxation studies of novel biomedical membranes will be continued. The possibility of applying STM/AFM to these systems will be investigated.

The solution structures of Ni, Zn and Cd forms of Desulforedoxin (small model protein) and 3 mutants will be determined. Experiments using ^{113}Cd to probe the H-bonding at the metal site will be carried out. Stabilisation of the enzyme ALAS in micelles will be attempted. TROSY type NMR experiments will be used to probe the structure in solution. The 3D structure of Heme Binding Protein will be determined.

NMR experiments in solution and the solid state will be carried out on statherin and statherin peptides with and without their biologically relevant hydroxyapatite (HAP) surfaces. The role of pH will be investigated. There is a strong correlation between the amount of phosphopeptides and the absence of dental caries.

On health related issues and biomaterials, the subject includes several interests either related to ongoing projects or to project applications the results of which are expected soon. Firstly, the use of NMR and MAS NMR to evaluate liver graft quality and compatibility as well as metabolism of donors and recipients will be pursued as the subject of an ongoing postdoctoral plan. Secondly, high resolution NMR and LC-NMR (equipment to be acquired in 2005) of biofluids will be carried out on selected plasma and bile samples towards different aims, respectively: characterise diabetes disorders in selected patients and attempt to use NMR for the early and objective diagnostic of several disorders found in foetuses and during pregnancy. This project involves collaboration of the Bissaya Barreto Maternity. Thirdly, work on the making of new biomaterials, based on a biopolymer/inorganic matrix, for use as implants and as drug carriers will be initiated.

On process and quality control in the beverages industry, using initial funding provided by UNICER to initiate work in 2005, work on the full chemical characterisation of selected beers by NMR and LC-NMR will be carried out as part of a doctorate plan. Aims include the use of the technique as a service for companies in order to aid in process control and in quality assessment.

The solid state NMR studies of solid sugars and their structural changes will be pursued, eventually including the study of dextran and cellulose in their interaction with peptides and other agents used to derivatise the polysaccharides in order to produce hydrogels.

Glass and Ceramic-Based Biomaterials. Research on the three systems $\text{SiO}_2\text{-CaO-P}_2\text{O}_5\text{-MgO}$, $\text{SiO}_2\text{-P}_2\text{O}_5\text{-CaO-MgO-K}_2\text{O}$ and $\text{SiO}_2\text{-Na}_2\text{O-MgO}$ will proceed with the purposes (1) and (2) previously established.

A new research project on mineralization studies in TiO_2 -containing glasses will start this year. Mineralization in silica-based glasses is highly dependent on the formation of Si-OH groups at their surface when immersed in simulated plasma. Ti-OH groups are also found at the surface of materials containing Ti. In the present project the behaviour in simulated plasmatic solutions of Si-containing glasses will be compared with the one of Ti-containing glasses.

We will pursue the modification of the morphology and composition of spray dried CaP granules by: (i) using suspensions of precipitated CaP particles with different morphologies and (ii) thermal treatment of the granules.

On the study of the loading and releasing behaviour of the spray dried porous granules for drugs and bioactive substances in solutions at physiological conditions, we will concentrate on the evaluation of the influence of the porosity of the material on the release profiles.

Scaffolds of chitosan /calcium phosphate with a regular macroporous microstructure and interconnected pores will be prepared by freeze-drying. The characteristics of composites prepared with different chitosan/calcium phosphate proportions will be evaluated and used to discuss the effects of the starting suspension composition on the microstructure properties of the final freeze-dried scaffold. The bioactivity of the scaffolds will be assessed.

CaP coatings on the surface of biodegradable polymers and polymer / CaP composites will also be produced.

Projects on the evaluation of in vitro activity of biomaterials and on polymer matrix composites for bone tissue engineering will be continued, and the recently approved (FCT-funded) set-up of a Laboratory of Biomaterials and Biomedical Devices effectively launched.

The refining of the process of crystallization and ageing of calcium phosphates in SBF solutions with $\text{CO}_2 / \text{HCO}_3^-$ buffer will be addressed and the influence of several trace metals in the crystallization and ageing of hydroxyapatite phases studied. Refining of the process of analysis of solid calcium phosphates by infrared spectroscopy will also be addressed, as well as the crystallization of magnesium phosphates and their ageing processes in physiological media.

PROCESS DEVELOPMENT AND OPTIMISATION

Phase Behaviour and Transport Properties Relevant in Environmental Protection, Chemical Processing and New Materials Production. New projects will start on developing the interfacial studies and the solubility of heavy hydrocarbons in water will address the problems of sea pollution due to hydrocarbon discharge. Another project will address the development of composite materials of paraffins for thermal insulation. Efforts will also be made concerning the development of a new improved model for the description of solid liquid equilibrium of paraffinic solutions and mixtures and the crystallographic characterization of molecules with long alkyl chains that may be of technologic interest.

The studies of the enhanced aeration of bioreactors using perfluorocarbon compounds will be pursued. We will in particular work with *Yarrowia lipolytica* and will extensively characterize the surface cell of this organism and the bioemulsifier produced by it. We will cultivate this yeast under different conditions of PFC content and agitation to optimize the aeration of

the reactor and enhance the production of lipase. Studies with *Trametes versicolor* will address the production of biopolymer and the treatment of paper pulps and effluents from the pulp and paper industries.

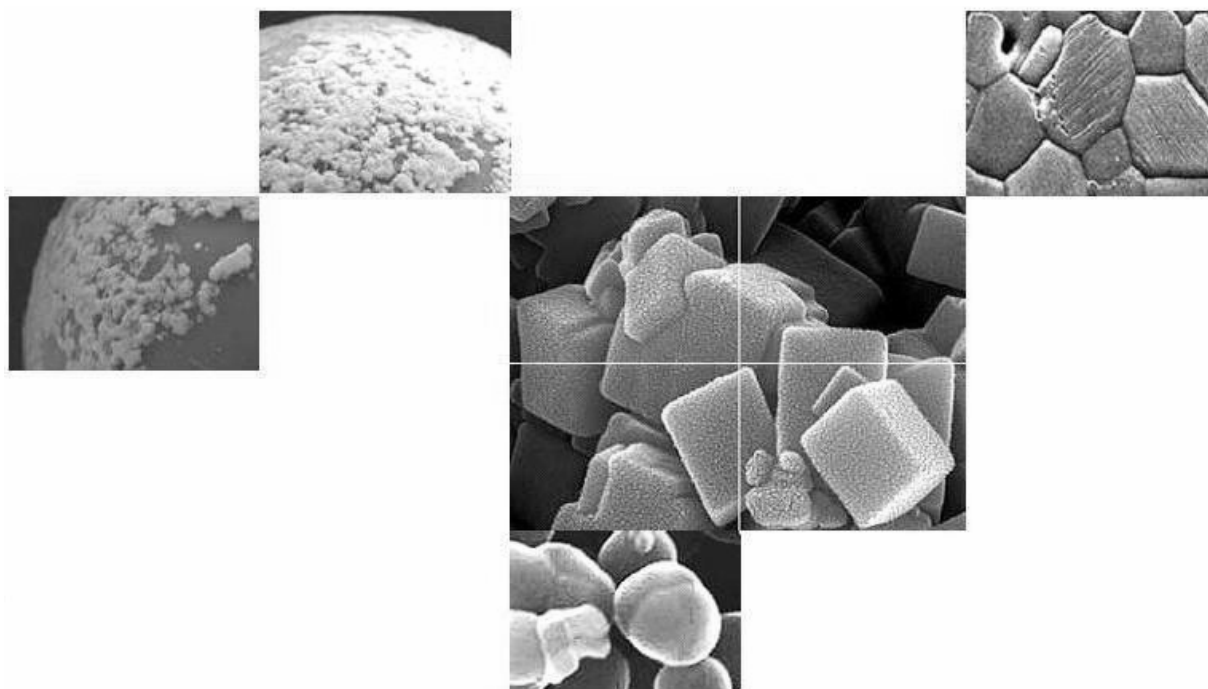
An important line of research will be the conversion of xylose to furfural in the presence of heteropoly acids. There is increasing demand for furfural in different fields, such as oil refining, plastics, pharmaceutical and agrochemical industries. Improvement of the chemical technology for the production of furfural therefore remains of great interest. In most industrial processes concentrated sulfuric acid is used as the catalyst, which presents numerous disadvantages. Our aim is to develop new catalytic processes for the transformation of pentosans/pentoses into furfural which offer environmental as well as economic benefits. In previous work, surfactant-templated micro-mesoporous silicas possessing sulfonic acid groups (SAGs) were found to be effective catalysts for the dehydration of D-xylose to furfural. However, it is not possible to recycle the solid acids (by washing) and maintain the high selectivities and conversions observed in the first runs. Thermal decomposition of the organic matter (coke) in the used catalysts is not possible due to the limited thermal stability of the surface-bound SAGs. In this respect, catalyst systems based on heteropoly acids (HPAs) may be a promising alternative. Future work will thus focus on Keggin-type HPAs of the type $H_3PM_{12}O_{40}$ ($M = Mo, W$) and $H_4SiW_{12}O_{40}$ as catalysts for the liquid phase dehydration of d-xylose to furfural. The kinetics of xylose conversion will be investigated in detail, varying either the reaction temperature or the initial concentrations of xylose and catalyst. In an effort to prepare heterogeneous catalysts, tungstophosphoric acid and Cs-tungstophosphoric acid will be supported on ordered mesoporous silicas. The pore size of the support and the catalyst loading will be fine-tuned in order to improve catalytic performance.

The parallel modelling of the PSA (pressure-swing-adsorption) problem will be finalized and the results presented. The enhancement the PC cluster will be addressed. A project on supercritical fluid extraction will be continued and another on membrane characterization for gas separation submitted for funding.

SECTION 4

SCIENTIFIC PRODUCTION

Until 31st December 2004



BIOMATERIAIS FUNCIONAIS PARA RECONSTRUÇÃO DE TECIDOS ÓSSEOS

ANDRADE A L

SUPERVISOR(S): DOMINGUES R Z AND FERREIRA J M F

UNIVERSITY OF MINAS GERAIS, BRASIL AND

UNIVERSITY OF AVEIRO, PORTUGAL

2004

DESENVOLVIMENTO DE METODOLOGIA DE FORMULAÇÃO DE MASSAS CERÂMICAS TRIAXIAIS UTILIZANDO DELINEAMENTO DE MISTURAS E OTIMIZAÇÃO

CORREIA S L

SUPERVISOR(S): SEGADÃES A M AND HOTZA D

FEDERAL UNIVERSITY OF SANTA CATARINA, BRASIL

2004

ESTUDO E MODELAÇÃO DA TENSÃO SUPERFICIAL E GELIFICAÇÃO DE COMBUSTÍVEIS PESADOS

QUEIMADA A J N

SUPERVISOR(S): MARRUCHO I M AND COUTINHO J A P

UNIVERSITY OF AVEIRO

2004

FABRICATION OF TIC MATRIX FE-AL AND NI-AL INTERMETALLICS COMPOSITES BY SPONTANEOUS MELT INFILTRATION AND INVESTIGATION OF MICROSTRUCTURE AND PROPERTIES OF THE COMPOSITES

GAO M-X

SUPERVISOR(S): PAN Y AND VIEIRA J M

UNIVERSITY OF ZHEJIANG, HANGZHOU, CHINA

2004

FLUÊNCIA DE POLÍMEROS-FENOMENOLOGIA E MODELAÇÃO DINÂMICA MOLECULAR

ANDRÉ J R S

SUPERVISOR(S): CRUZ PINTO J J C

UNIVERSITY OF AVEIRO

2004

MATERIAIS BASEADOS EM $LA(MG_{0,5}Ti_{0,5})O_3$ PARA APLICAÇÕES NAS MICROONDAS

SEABRA M P

SUPERVISOR(S): FERREIRA V M

UNIVERSITY OF AVEIRO

2004

OPTIMIZAÇÃO DAS CONDIÇÕES DE PROCESSAMENTO PARA INERTIZAÇÃO DE RESÍDUOS INDUSTRIAIS DE ALUMÍNIO EM MATRIZES CERÂMICAS ESTRUTURAIS

PEREIRA D A

SUPERVISOR(S): LABRINCHA J A; CASTRO F P AND

ALMEIDA M F

UNIVERSITY OF MINHO

2004

PREPARAÇÃO E CARACTERIZAÇÃO DE FILMES FERROELECTRICOS DE PZT PARA APLICAÇÕES ELECTROMECÂNICAS E EM MEMORIAS

PEREZ J

SUPERVISOR(S): KHOLKINE A AND VILARINHO P M

UNIVERSITY OF AVEIRO

2004

SÍNTESE E ESTUDO POLIOXOTUNGSTATOS DO TIPO KEGGIN COM INTERESSE

BALULA M S S

SUPERVISOR(S): CAVALEIRO A M V

UNIVERSITY OF AVEIRO

2004

SÓLIDOS MICROPOROSOS ZEOLÍTICOS COMO PRECURSORES DE NOVOS MATERIAIS LUMINESCENTES

MARQUES D A

SUPERVISOR(S): ROCHA J C M C G AND CARLOS L D

UNIVERSITY OF AVEIRO

2004

VALORIZAÇÃO DE RESÍDUOS INDUSTRIAIS E FORMULAÇÃO DE NOVAS COMPOSIÇÕES CERÂMICAS: REACTIVIDADE E COMPORTAMENTO TÉRMICO E ELÉCTRICO

RIBEIRO M J

SUPERVISOR(S): LABRINCHA J A AND FERREIRA J M F

UNIVERSITY OF AVEIRO

2004

CARACTERIZAÇÃO DA ESPERMATOGÉNESE SOB A ACÇÃO DE COMPOSTOS DE CHUMBO. ESTUDO EXPERIMENTAL NO RATINHO

GRAÇA A
SUPERVISOR(S): PEREIRA M L AND RAMALHO-SANTOS J
UNIVERSITY OF COIMBRA
2004

CARACTERIZAÇÃO E RECICLAGEM DE RESÍDUOS DERIVADOS DO CORTE DE ROCHAS NATURAIS

MANJATE R S
SUPERVISOR(S): FERREIRA J M F
UNIVERSITY OF AVEIRO
2004

CHEMICAL PREPARATION AND PROPERTIES OF CALCIUM PHOSPHATE BASED MATERIALS FOR BIOMEDICAL APPLICATIONS

SILVA M A M
SUPERVISOR(S): ALMEIDA M M T L AND COSTA M E V
UNIVERSITY OF AVEIRO
2004

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THE 17TH INTERNATIONAL SYMPOSIUM ON CERAMICS
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THE 7TH EUROPEAN CONFERENCE ON APPLICATIONS
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PHENOMENA INCLUDING RELATED EQUILIBRIUM PROCESSES
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AVEIRO, PORTUGAL
25-29 JUL 2004

X-RAY CHARACTERIZATION AND DOMAIN STRUCTURE OF
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SELF-FLUX SOLUTION METHOD

AMORÍN H; BDIKIN I K; SHVARTSMAN V V; COSTA M E
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THE 16TH INTERNATIONAL SYMPOSIUM ON
INTEGRATED FERROELECTRIC, ISIF 2004
GYEONGJU, KOREA
05-08 APR 2004

Congress Organisation

11TH INTERNATIONAL SYMPOSIUM ON SOLUBILITY
PHENOMENA – INCLUDING RELATED EQUILIBRIUM
PROCESSES

UNIVERSITY OF AVEIRO, PORTUGAL
25-29 JUL 2004

3^º ENCONTRO ASSOCIAÇÃO PARA O ENSINO DE
LABORATÓRIOS DE ENGENHARIA QUÍMICA - AELEQ

UNIVERSITY OF AVEIRO, PORTUGAL
02ND FEB 2004

7TH EUROPEAN CONFERENCE ON APPLICATIONS OF POLAR
DIELECTRICS

CZECH REPUBLIC
06-09 SEPT 2004

8TH EUROPEAN BIOLOGICAL INORGANIC CHEMISTRY
CONFERENCE - EUROBIC 8

PORTUGAL
02-06 JUL 2006

CONFERENCE ON SOLID STATE IONICS – TRANSPORT
PROPERTIES

PATRAS, GREECE
14-18 SEP 2004

COST E41 ACTION: "ANALYTICAL TOOLS WITH APPLICATION
FOR WOOD AND PULPING CHEMISTRY"

BRUSSELS, BELGIUM
7TH JUN 2004

COST E41 ACTION: "ANALYTICAL TOOLS WITH APPLICATION
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ESPOO, FINLAND
21-22 OCT 2004

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CHERBOURG, FRANCE
31 MAY-03 JUN 2004

XIX ENCONTRO DA SOCIEDADE PORTUGUESA DE QUÍMICA
UNIVERSITY OF COIMBRA, PORTUGAL

15-17 APR 2004

XXXIX REUNIÃO ANUAL DA SOCIEDADE PORTUGUESA DE
MICROSCOPIA ELECTRÓNICA E BIOLOGIA CELULAR

UNIVERSITY OF AVEIRO, PORTUGAL
4-6 NOV2004

Courses, Seminars and Training Programmes

ACÇÃO DE FORMAÇÃO PARA PROFESSORES E ESTAGIÁRIOS DE CIÊNCIAS E MATEMÁTICA SOBRE “O PROFESSOR E O DESENVOLVIMENTO DO CURRÍCULO – A ÁREA DE PROJECTO”

ESCOLA MARQUES CASTILHO, ÁGUEDA
17TH MAY 2004

CURSO "NANOSCALE CHARACTERIZATION AND PROPERTIES OF FERROELECTRICS"

UNIVERSIDADE "PRINCE OF SONGKLA", THAILÂNDIA
12-14 AGO 2004

ENCONTRO NACIONAL DE CIÊNCIA E TECNOLOGIA 2004 -
DINAMIZAÇÃO DE REDES TEMÁTICAS DE INVESTIGAÇÃO
INICIATIVA DO CONSELHO DOS LABORATÓRIOS ASSOCIADOS

UNIVERSIDADE DE AVEIRO
22 AND 23 OCT 2004

GEOPOLÍMEROS – DESENVOLVIMENTOS RECENTES E APLICAÇÕES NA ENGENHARIA

UTAD, PORTUGAL
19-20 NOV 2004

MEDIÇÃO E MODELAÇÃO DA FORMAÇÃO DE CERAS PARAFÍNICAS EM CRUDES A ALTAS PRESSÕES

CENPES, PETROBRAS
APR 2004

MEMBER OF THE ORGANISING COMMITTEE FOR THE ACTIVITIES OF THE SCIENCE & TECHNOLOGY WEEK AND PARTICIPATED IN THE VISITS TO THE CHEMISTRY DEPARTMENT

MEMBERS OF THE SCIENTIFIC COMMITTEE OF THE JMP (JOINT MASTER PROGRAMME) IN MATERIALS SCIENCE AND ENGINEERING

TECHNICAL UNIVERSITY OF HAMBURG/ HARBURG AND UNIVERSITY OF AALBORG
OCT 2004

PROJECTO E ESTÁGIOS DE ALUNOS DA LICENCIATURA EM QUÍMICA SOBRE PIGMENTOS E TINTAS EM COOPERAÇÃO COM A CIN – CORPORAÇÃO INDUSTRIAL DO NORTE. S.A

SEMINÁRIO SOBRE ARGAMASSAS DE CONSTRUÇÃO: DESENVOLVIMENTOS RECENTES E PERSPECTIVAS FUTURAS

UNIVERSIDADE DE AVEIRO
MAY 2004

SEMINÁRIO: APLICAÇÕES DA CITOMETRIA DE FLUXO NA BIOLOGIA, SAÚDE E AMBIENTE

UNIVERSIDADE DE AVEIRO
05 MAY 2004

SEMINÁRIO: NOVOS DESENVOLVIMENTOS DA ANÁLISE DE IMAGEM PARA APLICAÇÃO NA BIOLOGIA E SAÚDE

UNIVERSIDADE DE AVEIRO
25 MAY 2004

SEMINÁRIO: PERSPECTIVAS ACTUAIS DA REDUÇÃO DA FERTILIDADE MASCULINA

UNIVERSIDADE DE AVEIRO
25 FEB 2004

Reaching Out Activities

A ENG^a QUÍMICA E AS OUTRAS ARTES
COUTINHO J A P
ESC SEC LOURINHÃ, PORTUGAL
FEB 2004

ASSOCIATE EDITOR OF TRANSACTIONS ON ULTRASONICS,
FERROELECTRICS AND FREQUENCY CONTROL
KHOLKINE A
U S A
2004

AULA CONFERÊNCIA: NANOMATERIAIS
TRINDADE T
UNIVERSITY OF AVEIRO, PORTUGAL
05 APR 2004

AWARDED BY “ESTÍMULO À EXCELÊNCIA”
KHARTON V AND NAUMOVICH Y
2004

CIÊNCIA EM CASA (CANDIDATURA DO PROJECTO A
FINANCIAMENTO DA UNIÃO EUROPEIA ATRAVÉS DO SEXTO
QUADRO COMUNITÁRIO DE APOIO (ACTIVITY AREA
CALL: SCIENCE AND SOCIETY, SUPPORT SPECIFIC
ACTION: SCIENCE EDUCATION AND CAREERS))
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CICLO DA EDUCAÇÃO BÁSICA, PARA PROMOVER A
CIÊNCIA ATRAVÉS DE ACTIVIDADES ENVOLVENDO A
UNIVERSIDADE (CICECO E CENTRO DE GOVERNANÇA E
POLÍTICAS PÚBLICAS), AUTARQUIAS LOCAIS, ESCOLAS E
FAMÍLIAS
DE JESUS PEDROSA J D
2004

CO-EDITOR OF SPECIAL ISSUE OF THE JOURNAL OF SOLID
STATE ELECTROCHEMISTRY (SPRINGER) WITH
PROCEEDINGS OF THE OSSEP WORKSHOP “IONIC AND MIXED
CONDUCTORS: METHODS AND PROCESSES”
KHARTON V
ISSUE 9, VOLUME 8
2004

COLLABORATION WITH TWO LOCAL INDUSTRIES: FLEXIPOL
AND INDASA, VIA INDUSTRIAL INTERNSHIP WITH THE
PURPOSE OF ESTABLISHING FUTURE COLLABORATION
BARROS-TIMMONS A M
2004

COMUNICAÇÃO “REDES EMERGENTES DE COOPERAÇÃO
ENTRE INSTITUIÇÕES DE INVESTIGAÇÃO E EMPRESAS”
DE JESUS PEDROSA J D
ARRÁBIDA, PORTUGAL
12 OCT 2004

CONFERÊNCIA “LITERACIA E SISTEMA EDUCATIVO, COM
ESPECIAL ATENÇÃO À LITERACIA DAS CIÊNCIAS, DA
MATEMÁTICA E DA LEITURA”
DE JESUS PEDROSA J D
LISABON, PORTUGAL
21 DEC 2004

CONFERÊNCIA “AS UNIVERSIDADES E O DESENVOLVIMENTO
REGIONAL”
DE JESUS PEDROSA J D
UNIVERSITY OF TRÁS-OS-MONTES E ALTO DOURO,
PORTUGAL
24 MAR 2004

CONFERÊNCIA “CHANGING CONTEXTS AND CHALLENGES IN
EDUCATION”
DE JESUS PEDROSA J D
HIGH SCHOOL OF EDUCATION – INSTITUTO
POLITÉCNICO DE VIANA DO CASTELO
14 FEB 2004

CONFERÊNCIA “EDUCAÇÃO E FORMAÇÃO: REFLECTIR E
PROSPECTIVAR”
DE JESUS PEDROSA J D
12 FEB 2004

CONFERÊNCIA “MODOS E CONTEXTOS DE PRODUÇÃO E USO
DO CONHECIMENTO CIENTÍFICO”
DE JESUS PEDROSA J D
COIMBRA, PORTUGAL
19 NOV 2004

COUNCIL MEMBER OF ‘FERROELECTRICO DA SOCIEDADE
IEEE-UFFC’
KHOLKINE A
U S A
2004

DIRECTOR DOS CURSOS DE ENG^a CERÂMICA E DO VIDRO E DE
ENG^a DE MATERIAIS
SALVADO I M M
2004

“DOS OBJECTOS ESFÉRICOS À RODA GIGANTE (UMA
INTRODUÇÃO À QUÍMICA E USOS DOS
POLIOXOMETALATOS)”, PALESTRA DESTINADA A ALUNOS NO
ÂMBITO DO CICLO DE CONFERÊNCIAS ORGANIZADO PELO
NEQUA (NÚCLEO DE ESTUDANTES DE QUÍMICA DA
UNIVERSIDADE DE AVEIRO)
CAVALEIRO A M
UNIVERSITY OF AVEIRO, PORTUGAL
17 NOV 2004

M A LAVOISIER
XXII ENCONTRO JUVENIL DE CIÊNCIA
COUTINHO JAP
UNIVERSITY OF AVEIRO, PORTUGAL
SEPT 2004

MEMBER OF THE EDITORIAL BOARD OF THE JOURNAL OF
SOLID STATE ELECTROCHEMISTRY (SPRINGER)
KHARTON V
2004

NANOMATERIAIS E O DESENVOLVIMENTO DE NOVOS
PRODUTOS
TRINDADE T
UNIVERSIDADE DA BEIRA INETRIOR- COVILHÃ,
PORTUGAL
13 MAR 2004

NANOMATERIAIS: PEQUENAS PEÇAS PARA UM GRANDE
PALCO
TRINDADE T
ESCOLA SECUNDÁRIA FREI HEITOR PINTO-COVILHÃ,
PORTUGAL
10 MAR 2004

“O AMONÍACO. IMPORTÂNCIA E SÍNTESE INDUSTRIAL”
PALESTRA DESTINADA AOS PROFESSORES DO 11º ANO DAS
ESCOLAS DA REGIÃO
CAVALEIRO A M
ESCOLA SECUNDÁRIA ALVES MARTINS, VISEU,
PORTUGAL
13 DEC 2004

OLIMPÍADAS DE QUÍMICA
RIBEIRO-CLARO P J A
UNIVERSITY OF AVEIRO, PORTUGAL
MAR-MAY 2004

OLIMPÍADAS DE QUÍMICA JÚNIOR
RIBEIRO-CLARO P J A
UNIVERSITY OF AVEIRO, PORTUGAL
APR 2004

ORGANIZAÇÃO DE EXPERIÊNCIAS COM MATERIAIS EM
DIVERSAS ESCOLAS SECUNDÁRIAS DO DISTRITO DE AVEIRO
FRADE J R
2004

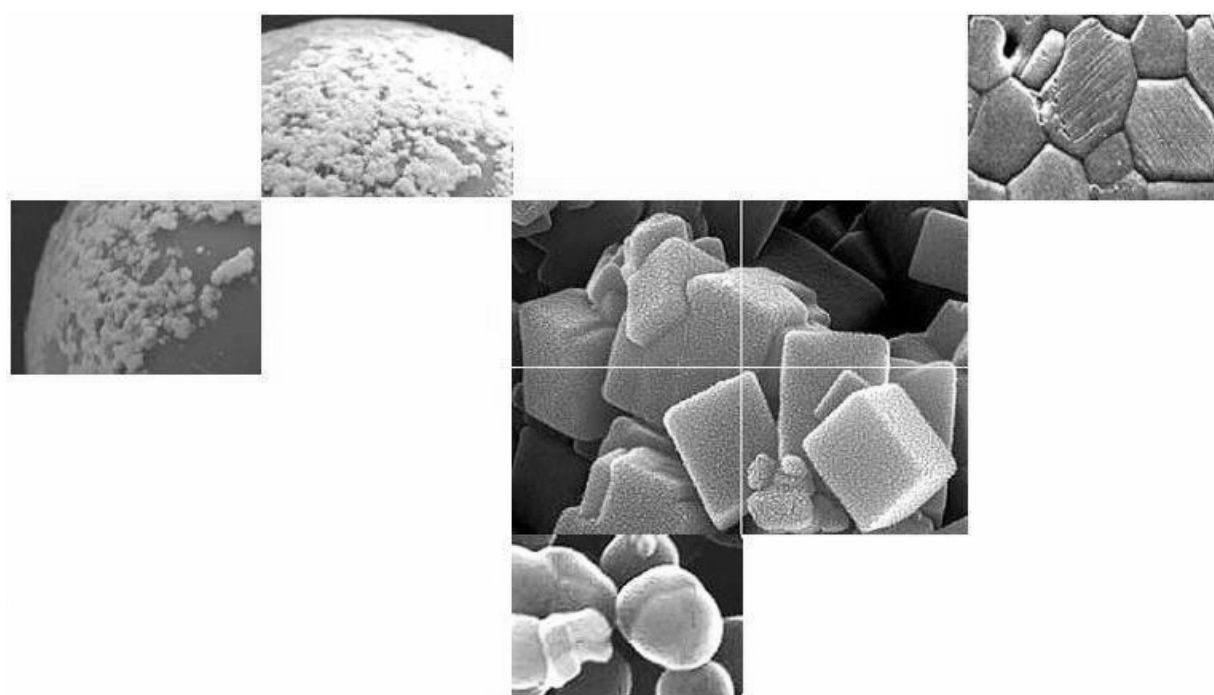
ORIENTAÇÃO DE NÚCLEOS DE ESTÁGIOS PEDAGÓGICOS NAS
ESCOLAS BÁSICAS E SECUNDÁRIAS
SANTOS T M
2004

PARTICIPAÇÃO NA SEMANA DA PRÁTICA PEDAGÓGICA

PRINCIPAIS PROBLEMAS DO ENSINO DA QUÍMICA EM
PORTUGAL- DOCUMENTO PREPARADO A PEDIDO DO SEC
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ROCHA J; RIBEIRO-CLARO P; MARTINS I; MARQUES G
AND FERRER CORREIA A J
AVEIRO, POTUGAL
2004

SECTION 5

PROJECTS



Projects Terminated

During 2004

CARACTERIZAÇÃO E RECICLAGEM DE LAMAS DERIVADAS DO CORTE DE ROCHAS NATURAIS E ORNAMENTAIS
PRAI-CENTRO

DEPOSIÇÃO DE DIAMANTE CVD SOBRE COMPÓSITOS CERÂMICOS NITRETO / CARBONETO DE SILÍCIO ($\text{Si}_3\text{N}_4/\text{SiC}$)
GRICES/CNPq4.3.1/2001

DESENVOLVIMENTO DE FORMULAÇÕES, PROCESSAMENTO E CARACTERIZAÇÃO DE MATERIAIS CERÂMICOS OBTIDOS A PARTIR DE RESÍDUOS INDUSTRIAIS
GRICES/CAPEs (4.1.3/CAPEs)

DEVELOPMENT OF COMPOSITES MADE OF CEMENT BINDERS AND LIGNOCELLULOSIC MATERIALS OF PORTUGUESE ORIGIN
POCTI/AGR/35480/1999

DOPAGEM ELECTRÓNICA E PROPRIEDADES FÍSICAS DE MANGANITAS COM VALÊNCIA MISTA
GRICES/CAPEs

ETUDES SOUS PRESSION ET TEMPÉRATURE DES TRANSFORMATIONS DE PHASE DES FULLERÈNES

GELS WITH INNOVATIVE OPTICAL, MAGNETIC AND ELECTROCHEMICAL PROPERTIES
POCTI/CTM/33653/1999

GROWTH AND CHARACTERIZATION OF $\text{SrBi}_2\text{Ta}_2\text{O}_9$ SINGLE CRYSTALS

HETEROGENISATION OF PHOTOCHEMICALLY AND CATALYTICALLY ACTIVE TRANSITION METAL COMPLEXES ON MESOPOROUS OXIDES
POCTI/32889/1999

IMMOBILIZATION OF SOLVENT-STABILIZED TRANSITION METAL COMPLEXES IN MICELLE-TEMPLATED SILICAS AND THEIR APPLICATIONS AS POLYMERISATION INITIATORS
CRUP, LUSO-GERMAN INTEGRATED ACTION TUM/UA (Proc. AI-A/03)

INTERACTIVE CALCIUM PHOSPHATE BASED MATERIALS PREPARED BY *POS*-HYBRIDIZATION AND IN *SITU* HYBRIDIZATION
POCTI/CTM/35516/1999

MAGNETOSTRUCTURAL MODULATION IN PEROVSKITE DERIVATIVES WITH STRONG ELECTRON CORRELATIONS
POCTI/CTM/35462/2000

NON-SYMMETRIC MACROCYCLES FOR METAL COMPLEXES AND SUPRAMOLECULAR AGGREGATES WITH PESTICIDES
POCTI/QUI/35396/99

PROCESSING, STRUCTURE AND DIELECTRIC PROPERTIES RELATIONSHIPS OF MICROWAVE CERAMICS
POCTI/CTM/40187/2001

REDE DE EXCELÊNCIA NA ÁREA DO PAPEL
PRAI-CENTRO

SÍNTESE POR COMBUSTÃO DE NITRETO DE ALUMÍNIO (AlN) E PROCESSAMENTO DE SUBSTRATOS DE AlN POR TAPE CASTING EM MEIO AQUOSO
PRAI-CENTRO

SPONTANEOUS INFILTRATION: AN ATTRACTIVE ALTERNATIVE FOR PROCESSING OF CMMC'S
GRICES/ MOST Proc. 4.13/ RP CHINA

STRUCTURAL AND FUNCTIONAL ASPECTS OF BULK AND INTERFACIAL POLYSACCHARIDE-SURFACTANT INTERACTIONS IN FOOD COLLOIDS
POCTI/BIO/33626/99

STRUCTURAL STUDIES OF CARBOHYDRATES IN THE SOLID STATE
POCTI/QUI/33075/2000

STUDIES OF COLOSSAL MAGNETORESISTIVE OXIDES WITH RADIOACTIVE ISOTOPES (IR)
CERN/FNU/49509/2002

STUDY OF THE VISCOELASTICITY PROPERTIES OF FLOURS: CORRELATION BETWEEN EXTENSIBILITY AND CHEMICAL COMPOSITION
B-9/03

STUDY OF THE CONTAMINATION DUE TO EXPLOITATION AND ABANDON OF MINES FROM PENEDONÓ AREA (VISEU)
POCTI/CTA/38579/2001

WASTE RECYCLING IN CERAMIC PRODUCTS
GRICES-CAPEs

Projects in Progress

ACER – ADVANCED CERAMIC MATERIALS: SYNTHESIS AND STRUCTURE
FP5 HPMT/CT/2000/00206

ADVANCED ELECTROCERAMICS: GRAIN BOUNDARY ENGINEERING
GRICES: COST 525

AERATION OF MULTIPHASE BIOLOGICAL REACTORS
POCTI/EQU/44427/2002

AROMA PERMEABILITY IN PACKAGING MATERIALS
POCTI/EQU/43356/2001

BIOLEARN - LEARNING FROM NATURE HOW TO DESIGN BIOMIMETIC ROUTES FOR PRODUCING CALCIUM-PHOSPHATE COATINGS ON POLYMERIC BIOMATERIALS
POCTI/CTM/38803/2002

BIOMATERIAIS DE Si_3N_4 REVESTIDOS A DLC E DIAMANTE CVD – BIODIAM
POCTI/CTM/45423/2002

CELLULOSE: SURFACE PROPERTIES, INTERACTION WITH BINDING DOMAINS AND ENZYMATIC MODIFICATION
POCTI/QUI/44368/2002

CHEMICAL BATH DEPOSITION OF BiVO_4 PIGMENTS
POCTI/QUI/46199/2002

COMBUSTION SYNTHESIS OF ONE DIMENSION ELONGATED α -SIALON CRYSTAL TO BE USED AS REINFORCING AGENTS FOR CMC PROCESSED BY COLOSSAL
POCTI/CTM/39419/01

CONTROLLING THE LENGTH SCALE THROUGH 'CHEMIE DOUCE': FROM INORGANIC FUNCTIONAL MATERIALS TO ORGANIC-INORGANIC HYBRIDS
POCTI/CTM/46780/2002

DEGRADATION MECHANISMS IN FERROELECTRIC MATERIALS VIA ATOMIC FORCE MICROSCOPY
423/DAAD

DESENVOLVIMENTO DE FERRAMENTAS EM METAL DURO REVESTIDAS POR FILMES MULTICAMADA Si_3N_4 /DIAMANTE LUSO-SPANISH INTEGRATED ACTION (E-70/03)

DESENVOLVIMENTO DE MÉTODOS EXPERIMENTAIS E TEÓRICOS PARA CARACTERIZAÇÃO DO COMPORTAMENTO TRIBOLÓGICO DE MATERIAIS CERÂMICOS E DE FILMES FINOS PARA APLICAÇÕES EM COMPONENTES MECÂNICOS, ESPECIALMENTE EM SELOS MECÂNICOS
GRICES/CAPEs (4.1.3/CAPEs)

DESENVOLVIMENTO TECNOLÓGICO APLICADO A MATÉRIAS-PRIMAS, PROCESSOS E PRODUTOS CERÂMICOS
GRICES/CAPEs

DESGASTE DE COMPÓSITOS DE MATRIZ DE ALUMÍNIO: INFLUÊNCIA DAS CONDIÇÕES DE PROCESSAMENTO E DE TRATAMENTO TÉRMICO
POCTI/CTM/46086/2002

DEVELOPMENT OF A BIOREACTOR-BASED CONNECTIVE TISSUE PRODUCTION LINE (TISSUE REACTOR)
CE / G5 RD – CT 2000 – 00282

DEVELOPMENT OF COMPOSITES MADE OF CEMENT BINDERS AND LIGNOCELLULOSIC MATERIALS OF PORTUGUESE ORIGIN
POCTI/AGR/35480/1999

DIELECTRIC PROPERTIES AND LATTICES DYNAMICS OF GRANULAR MATERIALS
POCTI/CTM/45284/2002

DOLCETA - DEVELOPMENT OF ON LINE CONSUMER EDUCATION TOOLS FOR ADULTS
EUCEN - B5-1000/03/000339

E-ECORISK - A REGIONAL ENTERPRISE NETWORK DECISION-SUPPORT SYSTEM FOR ENVIRONMENTAL RISK AND DISASTER MANAGEMENT OF LARGE-SCALE INDUSTRIAL SPILLS
EVG1-CT-2002-00068

EFFECT OF MAGNESIUM ON THE STRUCTURE AND SURFACE REACTIVITY OF SILICA-BASED GLASSES FOR BIOMEDICAL APPLICATIONS
POCTI/CTM/46251/2002

ENERGY TRANSFER FROM ELECTROLUMINESCENT POLYMERS TO PHOSPHORESCENT DOPANTS
POCTI/CTM/40063/2001

ESTUDO DO DIAGRAMA DE FASES DO C_{60} A ALTA PRESSÃO E TEMPERATURA
POCTI/CTM/40213/2001

HIGH-PRESSURE NMR SPECTROSCOPY OF POLYMERS AND BIOPOLYMERS IN CO₂ EMULSIONS
POCTI/QUI/42313/2001

IMMOBILIZED SURFACE RARE EARTH ARYLOXIDES. ACTIVATION OF SMALL MOLECULES (CO₂, CH₄, C₂H₆)
POCTI/QUI/42919/2001

IMPROVING THE YELD OF *EUCALYPTUS GLOBULUS* KRAFT PULP PRODUCTION: STRATEGIES, MECHANISMS AND IMPACT ON PULP ECONOMY
POCTI/EQU/46124/2002

INDUSTRIAL ECOLOGY: USUING SLUDGE FROM WASTEWATER TREATMENT PLANT OF SURFACE TREATMENT AS RAW MATERIAL FOR THE PRODUCTION OF REFRACTORIES
POCTI/CTA/42448/2001

INNOVATION AND SUSTAINABLE DEVELOPMENT IN THE FIBRE BASED PACKAGING VALUE CHAIN – SUSTAINPACK
FP6 2002 NMP/1 (IP 500311-2)

INTERACTIVE CALCIUM PHOSPHATE BASED MATERIALS PREPARED BY *POS*- HIBRIDIZATION AND IN *SITU* HIBRIDIZATION
POCTI/CTM/35516/1999

LABORATÓRIO DE BIOMATERIAIS E DISPOSITIVOS BIOMÉDICOS
CONC-REEQ/796/2001

MATERIAIS HÍBRIDOS PREPARADOS POR IRRADIAÇÃO
POCTI/CTM/44150/2002

MELHORIA DO RENDIMENTO E DAS CARACTERÍSTICAS FÍSICO-MECÂNICAS DAS PASTAS DE EUCALIPTO OBTIDAS PELO MÉTODO DO SULFITO ÁCIDO COM BASE DE MAGNÉSIO (CAIMA-INDÚSTRIA DE CELULOSE SA)
SIME 40/00905/1

MELHORIA DA BRANCURA DAS PASTAS DE EUCALIPTO OBTIDAS PELO MÉTODO DO SULFITO ÁCIDO COM BASE DE MAGNÉSIO
SIME 40/00905/2

MIXED CONDUCTING MEMBRANES FOR PARTIAL OXIDATION OF NATURAL GÁS TO SYNTHESIS GAS
NATO SIF 978002

MNAA - MATERIALS NETWORK FOR THE ATLANTIC AREA
INTERREG III

MODOS DE CRIAÇÃO E USO DO CONHECIMENTO CIENTÍFICO

MOLECULAR HYBRIDS OF POLYOXOMETALATES AND DIPOLAR ORGANIC MOLECULES: SYNTHESIS, CHARACTERIZATION AND DETERMINATION OF NON LINEAR OPTICAL PROPERTIES
POCTI/CTM/37713/2001

NANO COMPOSITES THICK FILM BY WET CHEMICAL PROCESSING
POCTI/CTM/44743/2002

NANOENGENHARIA DE PARTICULAS MAGNÉTICAS E LUMINESCENTES PARA TÉCNICAS DE SEPARAÇÃO E MARCAÇÃO DE CÉLULAS
3.64.33.7/NANOENGE.../CTS15

NANOPARTÍCULAS MAGNÉTICAS EM POLÍMEROS E HÍBRIDOS ORGÂNICO-INORGÂNICOS
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“NETWORK OF EXCELLENCE” ADVANCED MATERIALS ENGINEERING OF HYBRIDS AND CERAMICS (FAME)
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