Solid State

Manuel Leite de Almeida

The Solid State Group has developed during the past years a broad range of expertise in complementary areas of solid state science and focused, under a global approach, on selected new materials with unconventional electrical and magnetic properties. The nature of the group is multidisciplinary combining expertise ranging from the synthetic chemistry of either molecular materials with transition metal complexes or intermetallic compounds, to many different specialised solid state characterisation techniques. Among the more relevant facilities developed, operated by and maintained in the group, are a high temperature laboratory with crystal growth techniques (Czochralski, Bridgman and float zone), X-ray diffraction (single crystals and powder), EPR and Mössbauer spectroscopy, measurements of magnetisation by SQUID, Faraday and extraction techniques and AC-susceptibility, different electronic transport measurements and heat capacity, in a broad range of temperature and magnetic field.

The use of all these specialised characterisation techniques, often requiring low temperatures down to 0.3 K and high magnetic fields up to 18 T, lead to the development of facilities and expertise in cryogenics, unique in Portugal. The group was responsible for the installation at ITN in 1991 of a helium liquefier, which since then remained the only one operational in Portugal, serving also many users outside ITN. The replacement process of the old He liquefier by a new one, with expanded capacity and enhanced efficiency, was successfully completed in July after a 4 month period of interruption in the liquid helium supply.

The rare combination of the **preparative chemistry** expertise with the specialised **solid state physics** techniques enables the group to deal with different problems of modern materials science. Thanks to the valuable techniques developed, the group often act as a key partner of many national and international research projects. His activities have been centered in strategic selected type of materials, namely:

- Molecule based conducting and magnetic materials.
- Intermetallic compounds with *f*-elements.

Specific ongoing research projects are described in more detail in the following sections.

The group has an established tradition of research on **molecular conductors** since its origin at Sacavém. The two chain compounds, originally reported almost 3 decades ago, remained an important topic of research due to their unique properties. This research become now focused on a new fundamental aspects of crystalogenesis studied with AFM. The chemistry of bisdithiolene complexes and related TTF donors, namely substituted with coordinating groups as building blocks for multifunctional materials is a another topic of research that has been pursued.

The interests of the group have been extended during the last years to **molecular magnetism**. In the framework of the Network of Excellence ***MAGMANet*** the study of multifunctional switchable magnetic materials with possible conducting properties was developed and presently the combination of spin crossover compounds with other conducting and magnetic functionalities is being explored.

The research on **intermetallic compounds,** initiated in 1992 with emphasis on the study of phase diagrams of the type (f-element)-(d metal)-(p-element) and on new compounds with strongly correlated electronic behaviour and complex magnetic structures, has been recently extended to borides and thermoelectric materials.

The application of Mössbauer spectroscopy to different fields was pursued with large emphasis in the development of novel ferrite with mixed ionic-electronic conductivity for applications in alternative energy sources

A significant output of the group continued to be the **training and education of young scientists** which always plays a major role in its projects and the collaboration with Universities, namely in undergraduate courses.

Research Team

Researchers

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A. CERDEIRA, PhD Student, FCT grant.
M. HENRIQUES, PhD Student, FCT grant
B. VIEIRA, PhD Student, FCT grant
R. SILVA, BIC grant
S. OLIVEIRA, BIC grant
L. RIBEIRO, ITN grant (since July)

Collaborators

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Study of Ternary Phase Diagrams based on *f* and *d* elements

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Y. Verbovytskyy, P. A. Carvalho[1]*, A. Ferro*[1]*, O. Tougait*[2]*, H. Noël*[2]

Objectives

The major objective of this research line is to study (*f*-element)-(*d*-metal)-X ternary systems in order to establish the phase relations, and to identify, synthesise and characterise new ternary compounds. Basic information for the synthesis of pure samples and growth of single crystals is also expected to be obtained here.

Results

The work is focused on several unexplored or only partially explored ternary systems. Our previous results on the U-Fe-B system indicate the existence of five ternary borides, $UFeB_4$, UFe_2B_6, UFe_3B_2, UFe_4B and $U_2Fe_{21}B_6$, but it was not possible to obtain these phases as pure compounds and further studies were needed. Therefore, results of the *liquidus* projection for the B-rich corner were achieved, and in this region six ternary reactions were revealed. The two compounds belonging to it, $UFeB_4$ and UFe_2B_6, are confirmed to be formed by ternary peritectic reactions, but $UFeB_4$ has a considerably larger primary crystallization field, which points to an easier preparation of single crystals of this boride, when compare with UFe_2B_6. The study will be expanded in the near future to the remaining regions of this system.

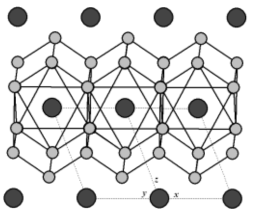
Ternary systems containing *f*-elements and zinc were almost not explored up to the beginning of our studies, with only some crystal structures of selected $R_xZn_yX_z$ compounds being reported.

The systematic investigation of the phase relations in the (*f*-element)–Zn–X systems was continued. The Yb-Zn-Ga and Eu-Zn-Al systems, in the 0-33.3 at.% R concentration range at 400°C, were completely explored and seven ternary compounds (the last six new) were found and their crystal structures were assigned:

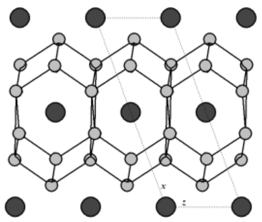
$YbZn_{0.75\text{-}2}Ga_{3.25\text{-}2}$, ($BaAl_4$-type),
$YbZn_{0.25–0.5}Ga_{3.75\text{-}3.5}$, ($CaCu_{0.15}Ga_{3.85}$-type),
$Yb_3Zn_{7.5–6.8}Ga_{3.5\text{-}4.2}$, ($La_3Al_{11}$-type),
$YbZn_{9.2\text{-}8.3}Ga_{1.8\text{-}2.7}$ ($BaHg_{11}$-type),
$Eu_2Zn_{13.9–14.3}Al_{3.1–2.7}$ (Th_2Zn_{17}-type),
$Eu_3Zn_{18}Al_4$ (Ce_3Zn_{22}-type) and
$EuZn_{1–2}Al_{3–2}$ ($BaAl_4$-type).

A relationship between the crystal structures existing along the "row" $CaGa_4$ ($YbGa_4$) – $CaCu_{0.15}Ga_{3.85}$ ($YbZn_xGa_{4\text{-}x}$) – $BaAl_4$ ($YbZn_xGa_{4\text{-}x}$) – La_3Al_{11} ($Yb_3Zn_{11–x}Ga_x$) has been established. The $CaGa_4$ and $CaCu_{0.15}Ga_{3.85}$ are distorted variants of the $BaAl_4$ structure type and the La_3Al_{11} structure can be obtained from $BaAl_4$ considering three unit cells and merging part of the 4*e* positions from this last structure type into the 2*d* positions of the La_3Al_{11} structure. An extended solid solution, $YbZn_{2–x}Ga_x$ ($0 \leq x \leq 1$), was also identified in the Yb–Zn–Ga system.

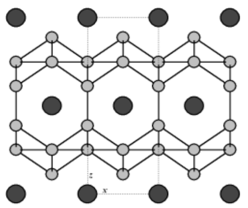
Preliminary results on the U-Fe-Ge and U-Fe-Sb isothermal sections were also obtained. These studies indicated a very rich U-Fe-Ge system, with more than 10 ternary phases (8 of them being new compounds) stable at 900ºC. In the U-Fe-Sb system only 2 phases, $UFe_{1\text{-}x}Sb_2$ and $U_3Fe_{1\text{-}x}Sb_4$ (both new), were identified until now.



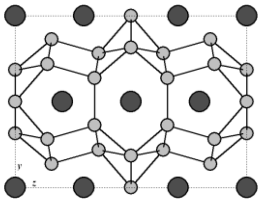
$CaGa_4$



$CaCu_{0.15}Ga_{3.85}$



$BaAl_4$



La_3Al_{11}

Published or in press work (selected)

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Yu. Verbovytskyy, A.P. Gonçalves, The Yb–Zn–Ga system: partial isothermal section at 400ºC with 0–33.3 at.%, *Intermetallics* **18** 655-665 (2010).

Y. Verbovytskyy, L.C. Alves, A.P. Goncalves, Phase relations of the Eu-Zn-Al system at 400 degrees C from 0 to 33.3 at.% Eu, *J. Alloys Compd.* **495** 39-44 (2010).

M.S. Henriques, D. Berthebaud, A. Lignie, O. Tougait, H. Noël, A.C. Ferro, A.P. Gonçalves, The study of the U-Fe-Ge system at 900ºC, *16th Workshop on Magnetism and Intermetallics*, IFIMUP-IN and Departamento de Física e Astronomia, Universidade do Porto, Porto, Portugal, 4 – 5 March, 2010, C3.

M.S. Henriques, A.C. Ferro, O.Tougait, H.Noel, A.P Gonçalves, The study of the isothermal section of the U-Fe-Sb ternary system at 700ºC, *17th International Conference on Solid Compounds of Transition Elements*, Annecy, France, September 5-10, 2010.

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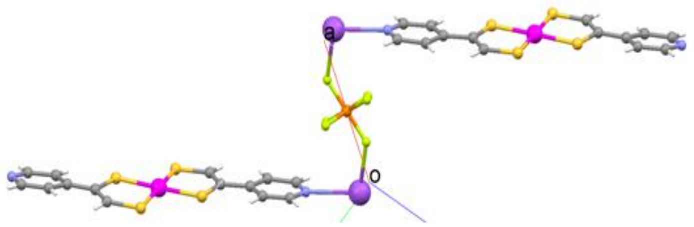
Neutral Pyridine-functionalized Bisdithiolene Complexes

S. Rabaça, A. Cerdeira, S. Oliveira, I. C. Santos, R. T. Henriques, M. Almeida,

Transition metal bisdithiolene complexes stable in their neutral state are not so frequent but they are currently attracting increasing interest due to a large potential interest as active components in optical, electrical and magnetic materials. Recently we have focused our attention on bisdithiolene complexes functionalized with pyridine groups which could coordinate other metals through the N atoms and lead to novel heterobimetallic magnetic networks. Under such effort the neutral complexes [Ni(4-pedt)$_2$] and [Au(4-pedt)$_2$] were prepared and characterised. The nickel complex was obtained as a single molecular compound while the gold one was obtained incorporated in a NaPF$_6$salt with, the pyridine nitrogen atom coordinating the sodium atom. None of these compounds is a good electrical conductor. The Ni complex is diamagnetic, but the paramagnetic gold complexes are connected only by short S$\cdots$S contacts in chains responsible for weak antiferromagnetic interactions.

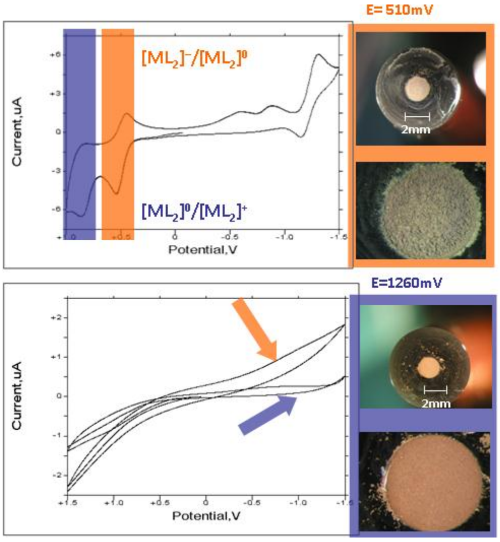


Electrodepositable Materials from Bisdithiolene Complexes Containing Pyridine Groups.

S. Rabaça, S. Oliveira, M. Afonso, M. Almeida

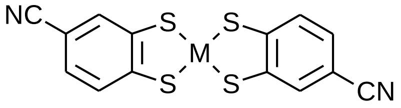
From the viewpoint of technological applications the processability of crystalline molecular materials is difficult, and conducting polymers have been more attractive for the development of technological devices. To fully utilize the properties of molecular species such as metal transition bisdithiolene complexes it is important to find ways of increasing the processability of these materials.

We have found that some transition metal bisdithioline complexes substituted with pyridine groups are excellent candidates to be used as precursors to electrodepositable materials. Our group have already develop a significant synthetic effort with pyridine substituted metal transition bisdithiolene complexes and within this project explored and study the better conditions and bisdithiolene precursor to be used as precursors of electrodepositable materials. The electrodeposition from solutions of anionic complexes [Au(4-pdddt)$_2$] , depending on the potential yields two different materials. In the range 0.51 V vs. Ag/Ag$^+$ a greenish deposit corresponding to the neutral complex was obtained, while above 1V it was formed a brownish deposit with metallic shine and higher conductivity corresponding to the cationic complex.



The series of transition metal cianobenzenodtiolate complexes [M(cbdt)$_2$]

A. Cerdeira, S. Rabaça, P. I. Girginova, I. C. Santos, L. C. J. Pereira, R. T. Henriques, D. Simão,[1] M. Almeida



The redox properties of dithiolene complexes are quite sensitive to the ligand nature and their relative contribution, compared to the transition metal, to the frontier orbitals. Following the recent synthesis in our group of the new ligand cbdt= 4-cyanobenzene-1,2-dithiolate an extended series of complexes [M(cbdt)$_2$] with different transition metals (M= Fe, Co, Ni, Au, Cu, Pd) could be obtained and characterised. These complexes were found to be mainly in the trans configuration except the Fe one which adopts a transoide dimmerised arrangement of cis-bisdithiolene [Fe(cbdt)$_2$] units. The redox behaviour of these complexes is intermediate between those based on unsubstituted benzenedithiolate (bdt) and dicyanobenezene (dcbdt). However at variance with some [M(dcbdt)$_2$] complexes no stable partially oxidised states could be obtained. The magnetic susceptibility of the [Co(cbdt)$_2$] complex is consistent with an undimerised arrangement of units in a high spin configuration. The use of these complexes as building blocks for conducting and magnetic materials and the secondary coordination through the N atom is currently being explored.

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Electrocrystalisation of the series of charge transfer salts (Per)$_n$[M (mnt)$_2$].

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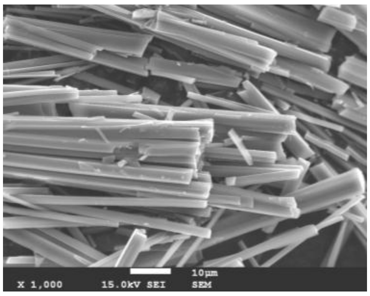

The series of charge transfer salts (Per)$_n$[M (mnt)$_2$] presents still unique features among molecular conductors calling for further studies requiring high quality single crystals. The electrocrystallisation has been in general a procedure with large success to obtain single crystals of many charge transfer salts. However the application of this technique to this series of perylene compounds has several limitations. Therefore a systematic study was initiated aiming at both obtaining higher quality single crystals and understanding the factor governing the crystallogenesis and determining the obtention of different phases.

Using high resolution AFM, performed both in-situ and ex-situ, the early stages of electrocrystallisation over different substrates were monitored and limiting nucleation and growth steps and mechanism could be identified. The systematic electrocrystallisation studies enabled for the first time (Per)$_2$[Pd(mnt)$_2$] single crystals to be obtained by electrocrystallisation. However these crystals were found to be mainly of the β-type semiconducting polymorph.

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Infrared properties of the one-dimensional organic conductors (perylene)$_2$ M (mnt)$_2$, M = Au, Pt.

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In spite of the large interest in the α–(perylene)$_2$[M(mnt)$_2$] compounds and extensive studies that have been performed in these compounds their optical properties have remained so far largely unexplored in part by the difficulty in obtaining suitable samples with large enough crystal faces. As a first step aiming at filling this lack of basic characterization, the optical properties of the one-dimensional organic conductors (perylene)$_2$[Pt(mnt)$_2$] and (perylene)$_2$[Au(mnt)$_2$] analogue have been investigated at ambient and low temperatures using the largest single crystals selected from several crystallization essays. The spectra with polarization along the molecular stacks show a simple Drude behavior with no detectable influence of electron-electron interactions. Based on the spectra for different polarizations an assignment of the vibrational modes was made. With polarization perpendicular to the stacks, a wide band observed in the mid-infrared range evidences a charge transfer between perylene and anion stacks. These electronic properties could be analyzed in comparison with other quasi-one-dimensional synthetic metals.

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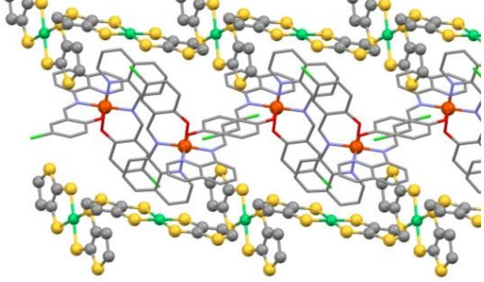
A new hybrid exhibiting room temperature spin-crossover and ferromagnetic cluster-glass behavior.

A. I. S. Neves, J. C. Dias, B. J. C. Vieira, I. C. Santos, M. B. C. Branco, L. C. J. Pereira, J. C. Waerenborgh, M. Almeida, D. Belo, V. da Gama

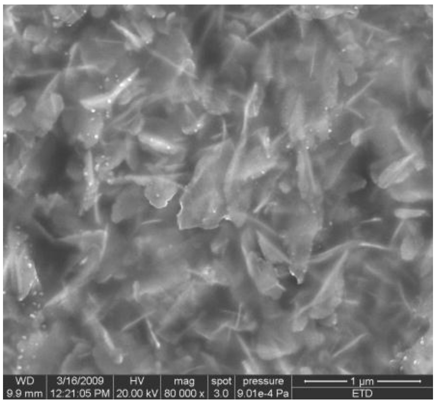
Spin-crossover (SCO) compounds have been systematically studied for more than 20 years but recently there was a renewal interest and the possibility of combining, in the same material, the SCO properties with other physical or chemical properties. In these context transition metal dithiolates appear as suitable anions to combine with SCO cations. Our experience on new dithithiophene complexes lead to the selection of the [Ni(α-tpdt)$_2$]$^-$ paramagnetic anion (tpdt= 2,3-thiophenedithiolate) and to a hybrid material, [Fe(5-Cl-qsal)$_2$][Ni(α-tpdt)$_2$]$^\cdot$CH$_3$CN (H5-Cl-qsal = N-(8-Quinolyl)-5- chlorosalicylaldimine) where the SCO phenomena is combined with the magnetic network of [Ni(α–tpdt)$_2$]$^-$. The crystal structure is based on alternated layers of [Fe(5-Cl-qsal)$_2$]$^+$ cations and of [Ni(α-tpdt)$_2$]$^-$ anions. The hybrid magnetic behaviour in this compound arises from the ferromagnetic cluster-glass behaviour, ascribed to the anions network, and from the spin crossover (SCO) of the [Fe(5-Cl-qsal)$_2$]$^+$ cations. The SCO process with $T_{1/2}$ = 298 K (high spin fraction = 0.5) was found to be rather sluggish, which is attributed to the effect of the anions that seem to restrict the structural distortions of the cations associated with the SCO process.



Single Component Molecular Conductors Processed as thin films

E. Laukhina,[1] V. Lebedev,[1] V. Laukhin,[1] A.P. Pino,[1] E.B. Lopes, A.I.S. Neves, D. Belo, M. Almeida, J. Veciana,[1] C. Rovira[1]

A simple procedure to cover a polycarbonate film with a metallic or a semiconductor-like layer of the single component conductor $[\mathrm{Au}(\alpha\text{-tpdt})_2]^0$ (tpdt=2,3-thiophenedithiolate) was established. These new organic bi-layer films are of technical interest due to the unusual combination of low crystallinity and high conductivity. This feature is unprecedented and an advantage within molecular conductor materials, whose electrical transport properties have been, so far, depended and directly controlled by their supramolecular structures. The low crystalline character of the $[\mathrm{Au}(\alpha\text{-tpdt})_2]^0$-based layer not only decreases the grain boundary resistance, allowing $[\mathrm{Au}(\alpha\text{-tpdt})_2]^0$ to behave as a metallic system, but also makes these low values of electrical resistance highly reproducible. The processing characteristics of polycarbonate films coated with a highly conductive $[\mathrm{Au}(\alpha\text{-tpdt})_2]^0$-based layer make them potentially useful for electronic applications where lightweight, large area coverage and flexibility are required (the figure shows SEM images from the metallic conducting layer of $[\mathrm{Au}(\alpha\text{-tpdt})_2]^0$ covering an organic polycarbonate film).



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Materials for solid oxide fuel cells and dense ceramic membranes

J C Waerenborgh, E. Tsipis, V. V. Kharton[1], A. Yaremchenko[1], E.N. Naumovich[1], M. V. Patrakeev[2], M.F.Lu[1,3]

Materials based on iron-containing oxides with perovskite-derived structures attract significant attention for energy-related electrochemical technologies, such as electrodes of solid oxide fuel cells or ceramic membranes for conversion of natural gas and biogas.

Transition metal-containing oxides are usually active for total combustion of hydrocarbons. High catalytic activities towards partial oxidation of methane have however been reported for perovskite-related ferrites. Explanations proposed for this phenomenon are: strongly-bonded lattice oxygen with low mobility, presence of carbonate species on the surface, or formation of catalytically active species such as iron carbide or metallic Fe. In order to obtain experimental information on the surface states of ferrite-based mixed conductors and on their role in CH_4 oxidation processes, which is still very scarce, a study by conversion electron Mössbauer spectroscopy started in 2010. The incorporation of Mo in $S_3Fe_2O_7$ was also investigated. A raise of the n-type electronic conductivity was observed although it remains insufficient to provide a high anodic performance in reducing atmospheres. On the other hand the incorporation of Mo leads to decreasing p-type charge carrier concentration and mobility, in agreement with Mössbauer data which showed an increased hole localization. Further enhancement of the dimensional stability and n-type electronic transport in $Sr_3(Fe,Mo)_2O_{7-\delta}$ is unlikely due to the relatively low Mo solubility, close to approximately 5% per f.u. in oxidizing conditions.

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Mössbauer spectroscopy in Materials Science

J.C. Waerenborgh, E. Tsipis

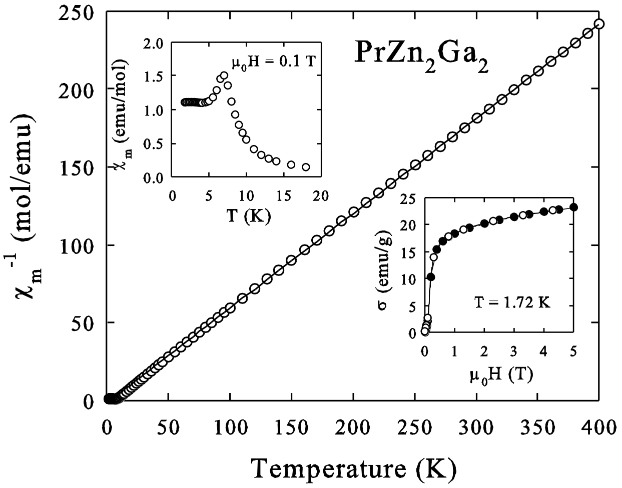
In 2010 the Mössbauer spectroscopy laboratory was kept running by one permanent and one post-doctoral researcher. This facility has been developed in ITN for 30 years and presently assists other national or foreign research institutions in the frame of joint research projects. In 2010 the application of the conversion electron technique in collaboration with CENIMAT/I3N, Faculdade de Ciências e Tecnologia , Universidade Nova de Lisboa is emphasized. This study allowed the evaluation of the structure, phase composition and SnO /SnO_2 ratio on p-type tin oxide thin film transistors prepared in different atmospheres and annealed at different temperatures. The high performance of the films prepared in CENIMAT will enable the production of fully transparent metal oxide semiconductors, associated with all the main advantages offered by transparent/oxide electronics. Other studies have also been performed such as characterization of the Fe-containing phases in environmental samples, a calcrete profile on the top of a calcareous consolidate dune, on multifunctional magnetic materials obtained by insertion of a spin crossover Fe^{III} complex into bimetallic oxalate-based ferromagnetic lattices, on UFe_{2+x} Laves phases prepared by ultrafast cooling in collaboration with the Univ. of Valencia, Spain; Charles University, Prague, Czech Republic; Dept. of Physics, Florida State University, USA.

Magnetic and strongly correlated electron behaviour in intermetallics

A.P. Gonçalves, J.C. Waerenborgh, L.C.J. Pereira, E.B. Lopes, M. Dias, M.S. Henriques, O. Sologub, S. Sério, M. Almeida, L. Havela[1], O. Tougait[2], H. Noël[2]

The understanding of the magnetic and strongly correlated electron behaviour of intermetallics containing *f*-elements, in particular the role of the *f*-orbitals, has been the major subject of a long-term project in the Solid State group.

The study of the U_2Fe_3Ge compound was continued in 2010 in order to identify the origin of the magnetic ordering. High-pressure X-ray diffraction measurements evidence no structural transition up to 25 GPa. Electrical resistivity measurements under pressure show that the Curie temperature decreases with the increasing pressure. The observed behaviour is similar to that observed in the $UNi_2$and UFe_2 Laves phases, pointing to an itinerant magnetism for U_2Fe_3Ge. Novel RZn_2Ga_2 (R= La, Ce, Pr, Nd, Sm) intermetallics have been synthesized and their crystal structures and magnetic properties were characterized. All compounds crystallize in the tetragonal $BaAl_4$ type structure. The $CeZn_2Ga_2$ compound remains paramagnetic down to 1.7 K but the Pr-, Nd- and Sm-based intermetallics order antiferromagnetically at low temperatures, with likely contribution of some ferromagnetic components.



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New materials for thermoelectric applications

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The identification, preparation and characterization of new thermoelectric systems with high figure of merit are of fundamental importance to their potential application in environment friendly energy applications. During 2010 we continued the studies on the family of electrically conducting glasses with general compositions in the range $Cu_{x+y}Ge_{20-x}Te_{80-y}$ ($0 \leq x \leq 20$; $0 \leq y \leq 10$) and started to explore the glasses in the Cu-Te-As system. Our preliminary results show that the melt spinning technique allows us to extend the Cu-Te-As glassy domain and leads to T_g values that permit the use of these glasses in applications up to 100°C. A maximum S^2/ρ value of $\sim 100 \mu W K^{-2} m^{-1}$ was obtained for the $Cu_{30}As_{15}Te_{55}$ composition, a power factor twice that of the best value obtained for the Cu-Ge-Te system and at a much lower cost of As compared to Ge. Further work is on the way to make bulk glass for these systems, more suitable for practical applications purposes.

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Superconductivity in TiNiSi-type structure compounds

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The coexistence of superconductivity and ferromagnetism in UGe_2, UIr, URhGe and UCoGe is an hot topic which raised the question about the dominant mechanism responsible for the pairing. Both URhGe and UCoGe belong to the uranium family of compounds with TiNiSi-type structure and it is expected that a similar behaviour can exist in analogous compounds.

Electrical transport measurements were performed on a UFeGe sample. Superconductivity was observed at T_C ~1K, with a critical field >4 T. However, SEM observations show that the sample has several phases and EDS measurements indicate Zr as impurity. Therefore, Zr-Fe alloys, which become superconductor below 1K, can be the reason for the observed superconductivity and further studies are need to determine the its origin.

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High magnetic field facility

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The ITN high magnetic field acility permits the study of electrical transport properties under magnetic fields with different orientations up to 18 T and for temperatures in the range 0.3-300 K, using both AC and DC techniques. During 2010 the 18T cryostat was used to pursue the research activities in different magnetotransport characterisation projects with emphasis on heavy fermion and non-Fermi liquid behaviour in intermetallics.

Several intermetallic compounds were characterized: In the continuation of the study of the interesting itinerant ferromagnet U_2Fe_3Ge this material was measured as a single crystal, and it was found a sharper transition in the resistivity at the Curie temperature T_C followed by a Fermi Liquid behaviour at low temperatures; $U_3Fe_4Ge_4$ which from the coefficient of T^2 dependence of the resistivity at low temperature was found to be a moderate heavy fermion; $U_9Fe_7Ge_{24}$ compound that is metallic, nonmagnetic and follows the expected Bloch-Gruneisen law; and some members of the $YbZn_xGa_{4-x}$ family of compounds that are also nonmagnetic and follow the expected Bloch-Gruneisen law for the resistivity at low temperatures.

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