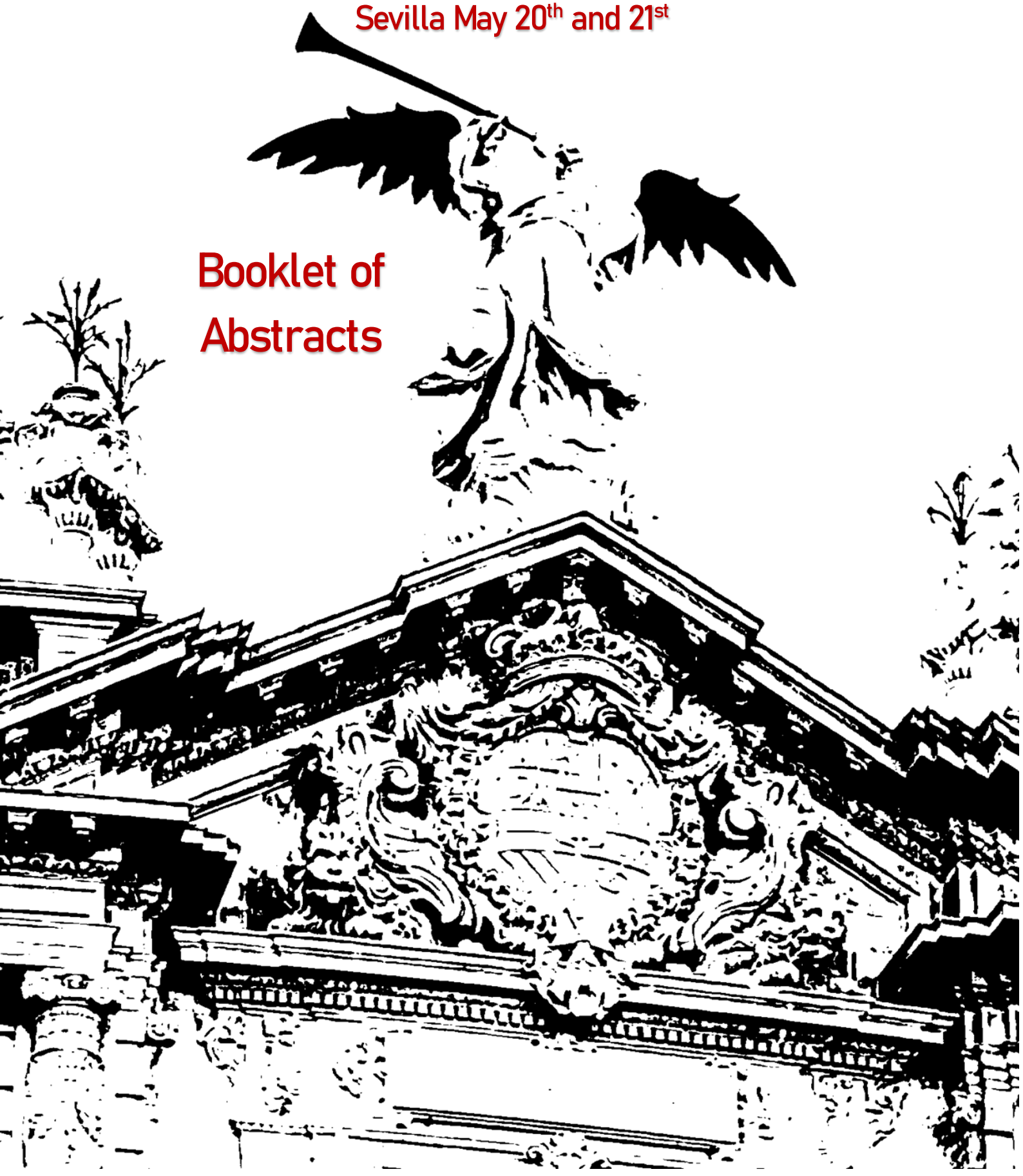
IBERMOSS 2024

3rd Iberian Meeting on Mössbauer Spectroscopy

Sevilla May 20th and 21st

Booklet of Abstracts



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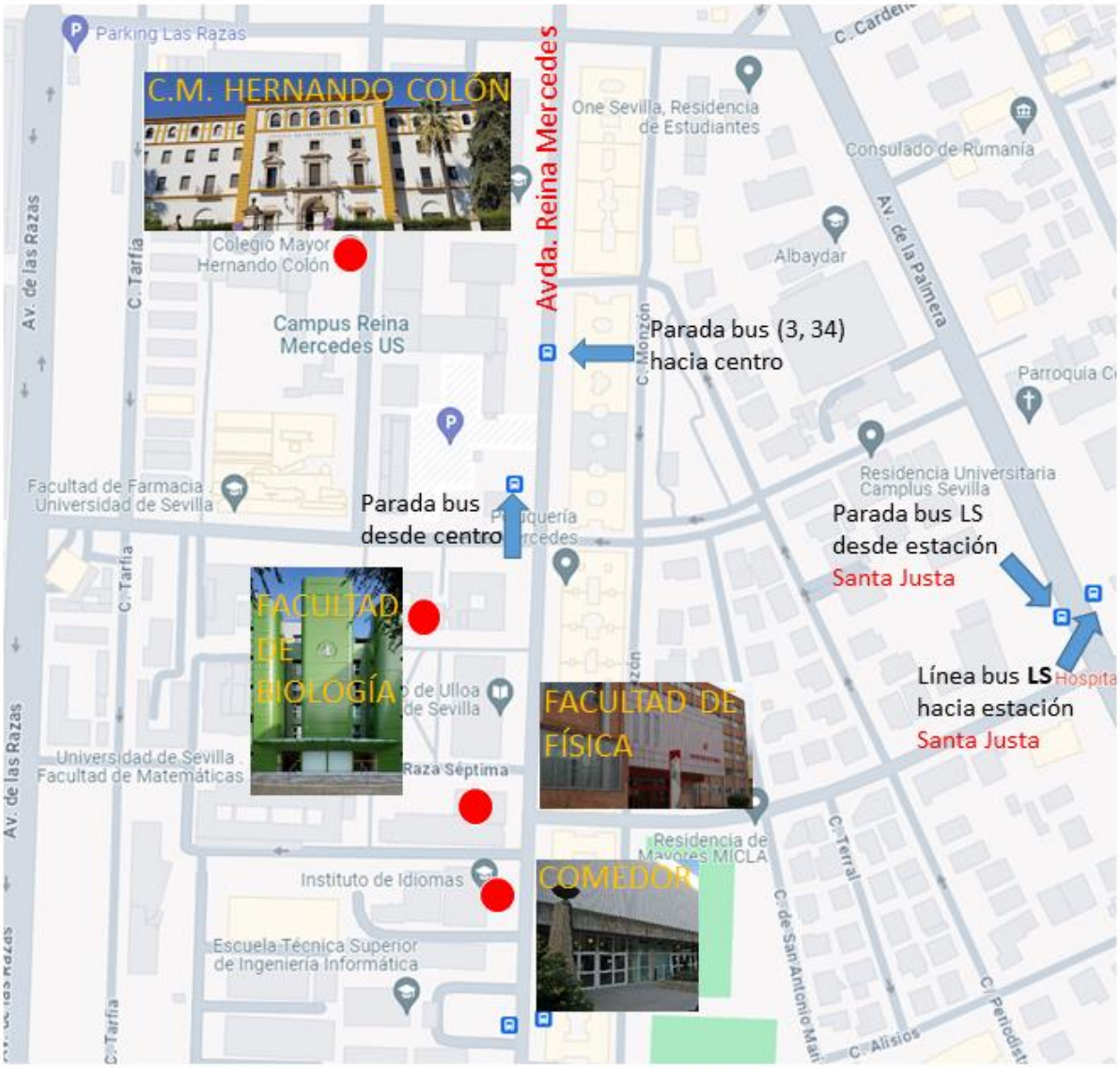
Maps



Restaurant “La Valiente” location and City Center

PROGRAM IBERMOSS2024

MONDAY MAY 20th

8:30-9:30	**REGISTRATION AT DESK**
9:30-10:00	**Welcome Speech;** Fernando Plazaola, Benilde Costa, Javier S. Blázquez
10:00-10:30	**Mössbauer Group at Bilbao: From the Starting to Yesterday;** J.A. García, J.S. Garitaonandia, D. Merida, **Fernando Plazaola**, I. Unzueta; *Zientzia eta Teknologia Fakultatea, Euskal Herriko Unibersitatea UPV/EHU, p.k. 644, 48080 Bilbao, Spain*
10:30-11:00	**Enjoying Mössbauer Spectroscopy during 45 Years; E. Molins**; *ICMAB – CSIC Campus UAB 08193 Bellaterra, Spain*
11:00-11:30	**COFFE BREAK**
11:30-12:00	**Mössbauer Spectroscopy as a Complementary Tool to the Study of Metallic Glasses; Pere Bruna**; *Departament de Física, Universitat Politècnica de Catalunya, BarcelonaTech (UPC), Av. Eduard Maristany 16, 08019 Barcelona, Spain*
12:00-12:30	**Mössbauer Spectroscopy at the IQF: A Tool for Almost Everything; José F. Marco**; *Instituto de Química Física Blas Cabrera, CSIC, Serrano 119, 28006 Madrid, Spain*
12:30-13:00	**From Mineralogy to Failure Analysis and Nanolayers; J. Rubín**; *Instituto de Nanociencia y Materiales de Aragón (INMA), CSIC-Universidad de Zaragoza, 50009, Zaragoza*

13:00-13:30	**^{57}Fe and ^{119}Sn Mössbauer Applications in the Study of Lithium- and Sodium-ion Battery Materials; José L. Tirado**, Ricardo Alcántara, Pedro Lavela, Carlos Pérez Vicente; *Departamento de Química Inorgánica e Ingeniería Química. Instituto Químico para la Energía y el Medioambiente. Universidad de Córdoba. Edificio Marie Curie. Campus de Rabanales 14071 Córdoba (Spain)*
13:30-15:00	**LUNCH**
15:00-15:30	**Capturing the Essence of Reaction Mechanisms Using Mõssbauer Spectroscopy (online); Pedro Tavares,** Alice Pereira; *UCIBIO-Applied Molecular Biosciences Unit, Department of Chemistry, NOVA School of Science and Technology, Universidade NOVA de Lisboa, Caparica, Portugal*
15:30-16:00	**From Instrumentation to Applications: Unveiling the Capability of Mössbauer Spectroscopy at IST/CTN (online); Bruno J.C. Vieira**, João C. Waerenborg; *Centro de Ciências e Tecnologias Nucleares, DECN, Instituto Superior Técnico, Universidade de Lisboa, Bobadela LRS, Portugal*
16:00-16:30	**Mössbauer Studies from Amorphous to Nanocrystalline Compounds; Jhon J. Ipus**, A. Vidal-Crespo, R. Caballero Flores, J.M. Borrego, J.S. Blázquez, C.F. Conde; *Dpto. Física de la Materia Condensada, Universidad de Sevilla, Spain*
16:30-17:00	**COFEE BREAK**
17:00-18:00	**57Fe Mössbauer Spectrometry Applied to Solid State Chemistry and Physics; Jean M. Greneche** (Conference at Main Hall of Faculty of Physics) *Institut des Molécules et Matériaux, Université du Mans, UMR CNRS, 72085 Le Mans Cedex, France*

19:00-20:00 Visit to "Fábrica de Tabacos" Main Building of the University

20:30 CONFERENCE DINNER AT RESTAURANT "LA VALIENTE"

TUESDAY MAY 21st

9:30-10:30	**Structural and Physical Properties of Fe Nanostructures; Jean M. Greneche;** *Institut des Molécules et Matériaux, Université du Mans, UMR CNRS, 72085 Le Mans Cedex, France*
10:30-11:30	**Nuclear Techniques for the Study of Matter. Positron Annihilation Spectroscopy and Neutron Scattering Techniques; Fernando Plazaola;** *Zientzia eta Teknologia Fakultatea, Euskal Herriko Unibersitatea UPV/EHU, p.k. 644, 48080 Bilbao, Spain*
11:30-12:00	**COFFEE BREAK**
12:00-12:20	**Optimizing Metallic Glasses: Investigating the Impact of Minor Additives Using Mössbauer Spectroscopy; Pere Bruna**, Joan Torrens-Serra**;** *Departament de Física, Universitat Politècnica de Catalunya, BarcelonaTech (UPC), Av. Eduard Maristany 16, 08019 Barcelona, Spain*
12:20-12:40	**Recycling of Hexaferrites;** Berja, C. Granados-Miralles, B. Podmiljsak, B. Saje, D. Huremović, T. Frangez, S. Kobe, J. E. Prieto, J. F.Marco, **J. de la Figuera**, J. Francisco Fernández, A. Quesada; *Instituto de Química Física Blas Cabrera, CSIC, Madrid 28006, Spain*
12:40-13:00	**Mössbauer Studies of Ni-Mn-Sn Based Metamagnetic Shape Memory Alloys; D. Mérida**, P. Lázpita, J. Gutiérrez, V.A. Chernenko, I. Unzueta, J.S. Garitaonandia, J.A. García, F. Plazaola; *University of Basque Country (UPV/EHU), Leioa 48940, Spain*
13:00-15:00	**LUNCH**
15:00-15:20	**Mössbauer and XPS Studies of β-hematin Crystals: Comparison of Two Synthesis Methods and Two Antimalarials;** L.J. Herrera, **J.F. Marco**, K.E. García, C.A. Barrero; *Instituto de Química Física Blas Cabrera, CSIC, Serrano 119, 28006 Madrid, Spain*

15:20-15:40	**Mössbauer Spectroscopy for Fe Electrodes in Fe-based Batteries and Single Atom Catalysts;** C. Alegre, N. Villanueva, L. Álvarez, D. Sebastián, H. A. Figueredo-Rodríguez, R. C. McKerracher, C. Ponce de León, M. J. Lázaro, **J. Rubín**; *Instituto de Nanociencia y Materiales de Aragón (INMA), CSIC-Universidad de Zaragoza, 50009, Zaragoza*
15:40-16:00	**Inversion Degree Determined by Mössbauer Spectroscopy: The Case of $(Mn_{0.2}Co_{0.2}Ni_{0.2}Cu_{0.2}X_{0.2})Fe_2O_4$ (X=Fe, Mg) Spinel High-Entropy Oxides Prepared Via Reactive Flash Sintering;** **A.F. Manchón-Gordón**, G.E. Almanza-Vergara, S. Molina-Molina, A. Perejón, J.S. Blázquez, P.E. Sánchez-Jiménez, L.A. Pérez-Maqueda; *Instituto de Ciencia de Materiales de Sevilla, ICMSE CSIC-Universidad de Sevilla, C. Américo Vespucio 49, Sevilla, 41092, Spain*

16:00 **CONCLUDING REMARKS**

ABSTRACT

Mössbauer group at Bilbao: from the Starting to Yesterday

J.A. García, J.S. Garitaonandia, D. Merida, **F. Plazaola**, I. Unzueta

Zientzia eta Teknologia Fakultatea, Euskal Herriko Unibersitatea UPV/EHU, p.k. 644, 48080 Bilbao, Spain

In this talk the Mössbauer group at Bilbao will be presented. The talk will show the reason why the first Mössbauer spectrometer was purchased. From this point on an historical development of the materials and techniques used will be given.

ABSTRACT

Enjoying Mössbauer Spectroscopy during 45 years

E. Molins

ICMAB – CSIC Campus UAB 08193 Bellaterra, Spain

Since my PhD, I have explored very different aspects of this spectroscopy. I begin with the calculations of the variation of the quadrupolar splitting ΔE_Q with temperature due to different populations of the electrostatic multiplets of Fe^{+2} in different compounds [1]. At that time, I built the third spectrometer of the Tejada's lab, the first one with a liquid nitrogen cryostat. We installed a Mössbauer spectrometer with He cryostat in the ICMAB's new building in 1992.

Working on Mössbauer spectroscopy is extremely pedagogical: the basics are in nuclear physics, but to go deeper into the technique, you must learn solid state physics (magnetism, crystal field, chemical bonds, phonons, electron delocalization…), material science, inorganic chemistry, radiation-matter interaction, among others. The instrumentation involves managing vibrations, cryogenics, high voltages, temperature measurement and control, high vacuum, electronics, signal processing, software of control, data treatment, radioactive protection, etc.

Some relevant examples from my experience will be shown. For instance, detecting $Fe^{+2.5}$ in mixed-valence compounds [2]. The possibility of using samples as sources, by doping with ^{57}Co. The miniaturization of the spectrometer, the MicroMössbauer [3], and the temperature warranty control. Mössbauer spectra as a key for understanding crystal structures: the case of the three-dimensional cubic phase $Ba_6Cu_{12}Fe_{13}S_{27}$ and that of the new mineral ferro-ferri-katophorite. Probe time and the superparamagnetism of small particles. The tuning of spin tunneling in the Fe_8 cluster [4]. We have also studied alloy nanoparticles, spin transitions, extraterrestrial materials and nanoparticles in porous materials for catalysis.

[1] E. Molins, A. Labarta, X. Tejada and V. Moreno. Electronic Structure Determination of Iron(II) complex with Inosine 5'-Mono-phosphate via Mössbauer measurements. *Zeits. für Physik B-Condensed Matter* 59 (1985) 419-422.

[2] Y. Portilla, I. Chávez, V. Arancibia, C. Loeb, J.M. Manríquez, A. Roig and E. Molins. Effect of Asymmetry on the Electronic Delocalization in Diiron and Iron-Cobalt Mixed Valence Metallocenic Compounds. Inorganic Chemistry 41 (2002) 1831-1836.

[3] L. Casas, E. Molins and A. Roig. Miniaturization of a Mössbauer Spectrometer using a Piezotransducer and a Solid State Detector. Hyperfine Interactions, 141 (2002) 125-129 and PCT WO 02/055972 A1.

[4] E. Molins, M. Gich, J. Tejada, J.M. Grenèche, F. Macià. Zero-field Quantum Tunneling Relaxation of the Molecular Spin in Fe8 observed by 57Fe Mössbauer Spectrometry. Europhysics Letters 108 (2014) 47004.

ABSTRACT

Mössbauer Spectroscopy as a Complementary Tool to the Study of Metallic Glasses

P. Bruna

Departament de Física, Universitat Politècnica de Catalunya, BarcelonaTech (UPC), Av. Eduard Maristany 16, 08019 Barcelona, Spain

Metallic glasses (MGs) are unique metallic alloys characterized by a disordered atomic structure, a result of rapid quenching from a molten state to solid, bypassing crystallization. This process leads to atoms arranged in a locally disordered manner, lacking the typical features found in crystalline materials such as defined atomic planes, grain boundaries, and crystalline defects.

The inherent disorder in their structure, coupled with the ease of compositional modification, renders MGs versatile for a wide array of applications. These include soft and hard magnetic materials, wear-resistant light alloys, biomedical implants or as catalysts for fuel-cell devices.

For making possible the transition from a laboratory material to to industrially viable materials is necessary to completely characterize the microstructure of MGs and understands its correlation with the macroscopic properties. It is at this stage where the use of Mössbauer spectroscopy becomes relevant as one of the few techniques that can yield information of disordered structures with only short-range ordering. Therefore, in the study of Fe-containing metallic glasses either in their as-quenched state (completely amorphous) or in an annealed state (partially o completely crystallized), Mössbauer spectroscopy emerges as a valuable complement to more conventional techniques like X-Ray Diffraction, Differential Scanning Calorimetry and/or magnetic measures.

In this presentation we will show a summary of some of the works done by the research group in the field of MGs highlighting all the aspects where Mössbauer spectroscopy gives us specific information difficult to obtain from other techniques like atomic quantification of amorphous and crystalline phases, identification of phases present at very low atomic concentrations, study of substitutional atoms in crystalline structures or magnetic texture between others.

ABSTRACT

Mössbauer Spectroscopy at the IQF: A Tool for Almost Everything

J.F. Marco

Instituto de Química Física Blas Cabrera, CSIC, Serrano 119, 28006 Madrid, Spain

There is a sentence attributed to the zoologist and Nobel Prize Konrad Lorenz that says: “Philosophers are people who know less and less about more and more, until they know nothing about everything. Scientists are people who know more and more about less and less, until they know everything about nothing.” This sentence, in fact, can be applied completely in both senses to scientists since there are two main, broad, ways to approach Science: to know everything about nothing or to know nothing about everything. The first approach would apply to those scientists who spend their entire careers studying the same system from many different points of view using multiple techniques. The second approach would apply to those scientists who are expert in a versatile technique which can be applied to many different systems. Many Mössbauer spectroscopists belong to this second classification and, in particular, this fits particularly well in the case of the Mössbauer laboratory of the IQF which has been operative for more than seven decades. The fact that ^{57}Fe is the “easiest” Mössbauer isotope is very fortunate since iron is a very important element in many scientific and technological disciplines as, for example, catalysis, corrosion, magnetism, biology and biomedicine, coordination chemistry, mineralogy, metallurgy, nanotechnology, coatings and thin films etc. The chemical, structural and magnetic information which Mössbauer spectroscopy can provide, sometimes in just a single shot, is crucial to understand many relevant phenomena occurring in the above mentioned fields. Along its history the Mössbauer lab at the IQF has explored tens of systems belonging to all those disciplines. In this talk we will focus in a few that have been important either for historical reasons related to our research group or for the relevance and impact they have had in the scientific literature. Thus, we will describe briefly with a few examples our research on corrosion of steel, covering both laboratory controlled experiments as well as in-field exposure tests, magnetite, both in nanoparticulate and thin film form and, to make reference to our more recent work, *strontium hexaferrite*.

ABSTRACT

From Mineralogy to Failure Analysis and Nanolayers

J. Rubín

Instituto de Nanociencia y Materiales de Aragón (INMA), CSIC-Universidad de Zaragoza, 50009, Zaragoza

Over the years the Mössbauer spectrometer at the Institute of Nanoscience and Materials (CIS-University of Zaragoza) has been used to contribute in the study of very diverse subjects. In this presentation I will show three very different cases: (i) The weathering products in mine wastes from abandoned tungsten and tin exploitations, in collaboration with a research group in mineralogy, (ii) The failure analysis of magnets in a servomotor, in collaboration with an electric motors company, and (iii) The study of Fe/Si multilayers with Conversion Electron Mössbauer Spectroscopy.

ABSTRACT

^{57}Fe and ^{119}Sn Mössbauer Applications in the Study of Lithium- and Sodium-Ion Battery Materials

J.L. Tirado, Ricardo Alcántara, Pedro Lavela, Carlos Pérez Vicente

Departamento de Química Inorgánica e Ingeniería Química. Instituto Químico para la Energía y el Medioambiente. Universidad de Córdoba. Edificio Marie Curie. Campus de Rabanales 14071 Córdoba (Spain)

Mössbauer spectroscopy (MS) of the quadrupolar nuclei ^{57}Fe and ^{119}Sn has multiple possibilities of application in the study of battery materials. The changes in oxidation state and chemical environment of the electroactive elements in the electrodes can be unfolded by this technique.

For Li-ion batteries (LIB), ^{57}Fe MS has been successfully applied in iron-based cathodes such as olivine $LiFePO_4$, and iron-substituted oxides [1]. For the anode, conversion electrode materials, and insertion phosphates [2] have given numerous examples. Regarding ^{119}Sn MS of LIB material, an outstanding application is to study of alloying and conversion-alloying anodes. Particularly, tin oxides, phosphates, and intermetallic compounds with other transition and non-transition metals [3].

In the case of the more recently developed Na-ion batteries (SIB), phosphate [4] and conversion halide [5] cathodes have been studied by ^{57}Fe MS. For the SIB anode, conversion oxides, and chalcogenides have been characterized by ^{57}Fe MS [6]. Finally, Na-Sn alloys and conversion-alloying compounds have also been studied by ^{119}Sn MS [7].

In this communication, we summarize the most relevant findings in the studies carried out in this field by our research team (FQM288).

[1] R. Alcántara et al., in Mössbauer Spectroscopy: Applications in Chemistry, Biology, and Nanotechnology, V. K. Sharma, G. Klingelhöfer, T. Nishida (Eds.). J. Wiley & Sons, Inc. (2013) Ch. 28, and references therein.
[2] C. Vidal-Abarca et al. J. Mater. Chem. 22 (2012) 21602.
[3] R. Alcántara et al. Hyperfine Interact. 187 (2008) 13–17.
[4] M.J. Aragón et al. J. Electrochem. Soc. 162 (2015) A3077-A3083.
[5] L.T. López-Ch et al. J. Alloys Compd. 968 (2023) 172123.
[6] S. Rubio et al. ACS Appl. Energy Mater. 3 (2020) 10765−10775.
[7] S. Rubio et al. J. Clean. Prod. 359 (2022) 131994.

ABSTRACT

Capturing the Essence of Reaction Mechanisms Using Mõssbauer Spectroscopy

Pedro Tavares, Alice Pereira

UCIBIO - Applied Molecular Biosciences Unit, Department of Chemistry, NOVA School of Science and Technology, Universidade NOVA de Lisboa, 2829-516 Caparica, Portugal

Mössbauer spectroscopy shines in characterizing the solution structure of iron cofactors, their oxidation and spin states, and certain molecular interactions. It offers valuable insights into how a macromolecule interacts with substrates, inhibitors, and partners in metabolic pathways. However, this information represents a static picture, typically capturing an enzyme's "ready state" or a cofactor bound to a ligand. Understanding the intricate dance of molecules in reaction mechanisms requires an additional dimension: time.

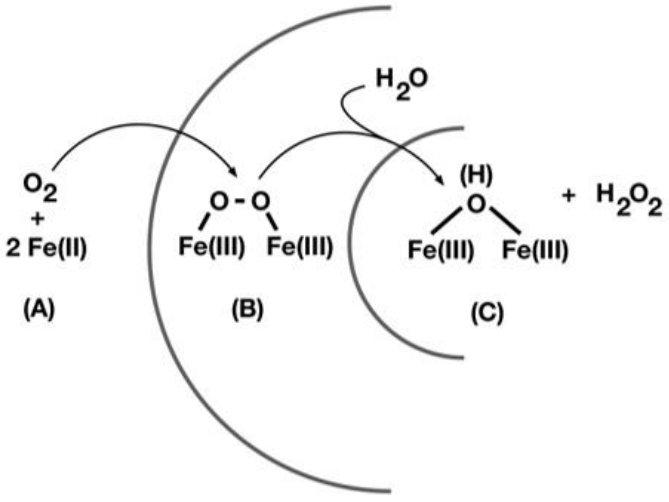


Figure 1: Representation of the molecular mechanism proposed for the fast ferroxidation process in eukaryotic ferritins

[1] P. Tavares, A.S. Pereira, Fast Kinetics—Stopped-Flow and Rapid Freeze-Quench. In: Pereira, A., Tavares, P., Limão-Vieira, P. (eds) (2019) Radiation in Bioanalysis. Bioanalysis, vol 8. Springer, Cham. https://doi.org/10.1007/978-3-030-28247-9_13

ABSTRACT

From Instrumentation to Applications: Unveiling the Capability of Mössbauer Spectroscopy at IST/CTN

Bruno J.C. Vieira, João C. Waerenborgh

Centro de Ciências e Tecnologias Nucleares, DECN, Instituto Superior Técnico, Universidade de Lisboa, E.N. 10, km 139.7, 2695-066 Bobadela LRS, Portugal

This presentation will offer an overview of the research conducted at the Mössbauer Spectroscopy Laboratory of the Instituto Superior Técnico (IST)/ Campus Tecnológico e Nuclear (CTN). Following a general introduction to the laboratory's main scientific areas of interest, a detailed description of the available facilities will be presented. Additionally, current challenges associated with source acquisition and licensing will be referred. Examples of significant results achieved within each area will be presented, highlighting the laboratory's scientific contributions.

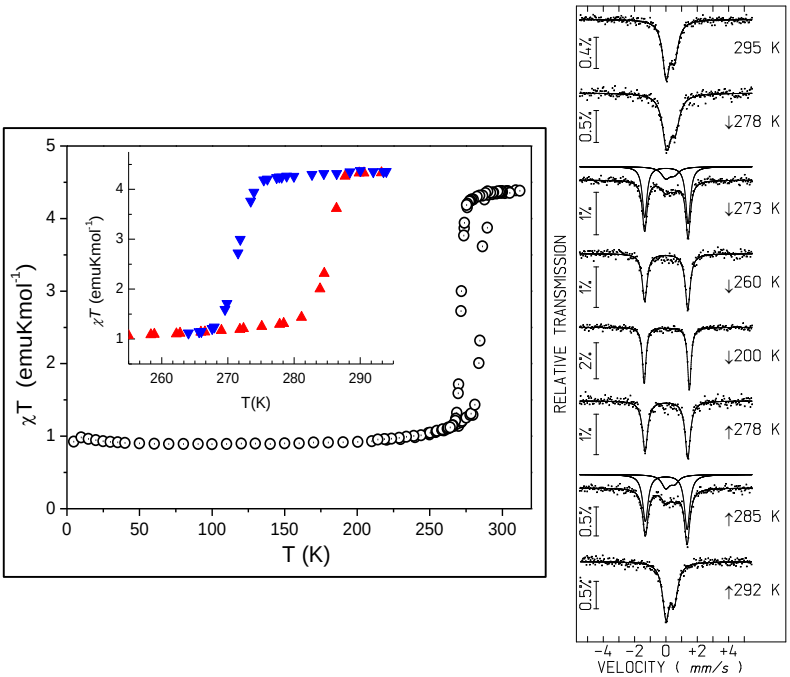


Figure 1: Magnetic characterization of an iron complex exhibiting spin crossover transition associated with hysteresis (left) and the corresponding Mössbauer spectra evidencing the transition (right) [1].

[1] B.J.C. Vieira, J.C. Waerenborgh, et al., Inorg. Chem. 54 (2015) 1354–1362.

ABSTRACT

Mössbauer Studies from Amorphous to Nanocrystalline Compounds

J.J. Ipus, A. Vidal-Crespo, R. Caballero-Flores, J.M. Borrego, J.S. Blázquez, C.F. Conde

Dpto. Física de la Materia Condensada, Universidad de Sevilla, Spain

Mössbauer spectroscopy was introduced in University of Sevilla lab to study the Fe environments in amorphous and nanocrystalline soft magnetic materials [1-3]. Several works have demonstrated Mössbauer spectroscopy as a more sensitive tool than x-ray diffraction to detect small fractions of crystallization products [4]. Concerning mechanically alloyed systems, it was observed that the evolution of hyperfine parameters of nanocrystalline samples could be rescaled for different milling processes using a Ω^3 law, where Ω is the rotational frequency of the mill [5]. Mössbauer technique also allows us to extract information about the concentration of immiscible elements into the Fe lattice in supersaturated solid solutions [6,7].
Beyond the characterization of amorphous and nanocrystalline materials, Mössbauer is also used to study the microstructure and phase transitions on intermetallics, shape memory and high entropy alloys [8-11].

[1] J.J. Ipus et. al. Intermetallics 15 (2007) 1132
[2] J.J. Ipus et. al. Intermetallics 16 (2008) 1073
[3] J.S. Blázquez et. al. J. All. Comp. 469 (2009) 169
[4] J.J. Ipus et. al. Intermetallics 18 (2010) 565
[5] J.J. Ipus et. al. Intermetallics 16 (2008) 470
[6] J.S. Blázquez et. al. J. All. Comp. 610 (2014) 99
[7] J.J. Ipus et. al. Intermetallics 49 (2014) 98
[8] J.S. Blázquez et. al. J. All. Comp. 646 (2015) 101
[9] A. Vidal-Crespo et. al. Metals 9 (2019) 534
[10] A.F. Manchón-Gordón et. al. J. All. Comp. 844 (2020) 156092
[11] L. Santiago-Andrades et al. Nanomaterials 14 (2024) 27

ABSTRACT

^{57}Fe Mössbauer Spectrometry Applied to Solid State Chemistry and Physics

J.M. Greneche

Institut des Molécules et Matériaux du Mans, UMR CNRS 6283, Le Mans Université, 72085 Le Mans Cedex, France.

The aim of this presentation is to give some basical tools demonstrating that the ^{57}Fe Mössbauer spectrometry is a very useful technique bringing relevant information on the chemical and physical nature of Fe-species containing compounds.
After a few brief comments on the fundamental aspects of the Mössbauer effect and the origin and the physical significance of the hyperfine interactions, we address then some useful and necessary advices on the instrumental aspects (particularly how to optimize the samples) and the fitting procedures of Mössbauer spectra. Indeed, this step which is crucial requires great knowledge on the hyperfine interactions and on the sample. These different points are crucial to make ^{57}Fe Mössbauer spectrometry suitable to investigate any Fe-containing materials as crystalline, amorphous, nanocrystalline and (nano)composites in different areas such as chemistry, physics, metallurgy, corrosion, magnetism, mineralogy, earth science, soils, biology, archeology, …
So, the purpose of the presentation aims to illustrate first the large relevance of this local probe and non-invasive technique for understanding the chemical composition and structure, the phase transitions, the magnetic structures and their respective evolution versus external stimuli, in complementary to diffraction techniques, electron microscopies and magnetic measurements to establish the correlation between structure and physical properties. The examples selected are concerned by iron fluorides, iron oxides (ferrites), metal organic frameworks and metallic crystalline and amorphous alloys. We also show that the Mössbauer measurements have to be performed in some cases as a function of temperature and external magnetic field to make unique the description of the hyperfine structure. In addition, computer modelling of hyperfine parameters based on *ab initio*, Monte Carlo and molecular dynamics approaches could be very useful to compare with experimental results.

ABSTRACT

Structural and Physical Properties of Fe Nanostructures

J.M. Greneche

Institut des Molécules et Matériaux du Mans, UMR CNRS 6283, Le Mans Université, 72085 Le Mans Cedex, France.

After discussing the main characteristics of artificial and natural nanostructures, including nanoparticles, multilayers, and nanostructured materials, we review the questions that need to be resolved in order a better understanding their structural and physical properties. The first step is to establish correlations between their physical properties and their size, the morphology and chemical composition of the crystalline grains, the thickness of the grain boundaries and the quality of the interfaces, respectively. The second step is to distinguish the nature of surfaces and grain boundaries and to understand their role on the magnetic properties.

To illustrate this strategy, we give some examples based on results obtained by ^{57}Fe Mössbauer spectrometry, a local probe technique well appropriate to the study of these complex Fe-containing nanomaterials. We show that this fundamental approach was necessary to better understand the relationship between the morphology of nanostructures and their physical and chemical properties and also to characterize some natural and highly complex nanostructures resulting from corrosion science, geology, environmental science, archeology, agriculture.

ABSTRACT

Nuclear Techniques for the Study of Matter. Positron Annihilation Spectroscopy and Neutron Scattering Techniques

Fernando Plazaola

Zientzia eta Teknologia Fakultatea, Euskal Herriko Unibersitatea UPV/EHU, p.k. 644, 48080 Bilbao, Spain.

Within a Mössbauer spectrometry meeting, focused on discussing material problems that can be evaluated by such a technique, the title of the presentation aims to give an overview of some other nuclear techniques, complementary to Mössbauer spectrometry, that can provide valuable information to solve different unknown problems in materials. Therefore, both nuclear techniques will be briefly introduced and then it will be shown how they can be used to explain problems in materials characterization. In particular, their application will focus mainly on magnetic shape memory alloys, as they are an interesting class of smart materials, which the Bilbao group has studied in depth.

Among the wide range of characterization tools available, neutron scattering techniques are used to gain advanced knowledge of the structure and magnetism of magnetic shape memory alloys. A comprehensive review about the characterization of these smart materials using neutron techniques will be presented.

The potential of positron annihilation spectroscopy, a nuclear technique well suited to study open volume defects in materials, such as vacancy-type defects, will be demonstrated, using it to explain unexpected behaviour appearing in magnetic shape memory alloys.

ABSTRACT

Optimizing Metallic Glasses: Investigating the Impact of Minor Additives Using Mössbauer Spectroscopy

Pere Bruna[1], Joan Torrens-Serra[2]

[1] *Departament de Física, Universitat Politècnica de Catalunya, BarcelonaTech (UPC), Av. Eduard Maristany 16, 08019 Barcelona, Spain.*

[2] *Departament de Física, Universitat de les Illes Balears, Cra. De Valldemossa km 7.5, 07122, Palma de Mallorca. Spain.*

The use of Fe-based bulk metallic glasses has primarily targeted the substitution of crystalline soft magnets. However, the challenge in manufacturing parts with the desired shape has impeded their widespread industrial adoption. To enhance the glass-forming ability of these alloys, one approach involves incorporating minor elements, particularly rare-earths [1,2]. However, such additions can alter their crystallization behavior, consequently influencing their ultimate properties. Following annealing of these glasses, the predominant phases formed are intricate large unit cell crystalline structures like the $Fe_{23}B_6$ phase, which suppress long-range atomic rearrangements and stabilize the supercooled liquid state. Mössbauer spectroscopy emerges as a particularly suitable experimental technique for characterizing both the primary crystalline phase and the remaining amorphous matrix in Fe-containing glasses. In this work we present a review of the effect of hyerarchically add minor additions of different elements to the base-alloy $(Fe_{0.6}Co_{0.4})_{0.75}Si_{0.05}B_{0.20}$ [3,4]. Firstly we analyse the changes induced by the addition of Mo, Nb and Zr and, afterwards, the rare-earths Y and Gd. The interplay between the glass forming ability of the alloys and their macroscopic properties as a function of the additions have been studied by means of Mössbauer spectroscopy, allowing us to fully characterize the crystallization path of these alloys. The different observed microstructures have been correlated with complementary measures performed by X-ray diffraction and with studies of their thermal and chemical stability and magnetic properties.

[1] W.H. Wang. Prog. Mater Sci 52 (2007) 540.
[2] J. Li, W. Yang, M. Zhang, G. Chen, B. Shen. J. Non-Cryst. Solids 365 (2013) 42
[3] J. Torrens-Serra, P. Bruna, M. Stoica and J. Eckert. Journal of Alloys and Compounds 704 (2017) 748-759
[4] S.L. Panahi, P. Ramasamy, F. Masdeu, M. Stoica, J. Torrens-Serra, P. Bruna. Metals 11 (2021) 1293.

ABSTRACT

Recycling of Hexaferrites

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The most used materials by wieght in the world for the fabrication of permanent magnets are strontium and barium hexaferrites [1,2]. In the coming years it is expected that their demand will increase several times due to the speeding pace of the decarbonization of the economy, where magnets play a crucial role in energy generation and motion. Thus the issue of the possible role of recycling of such materials will be central in the near future, allowing for a reduction in their production costs as well as a diminishing impact on the environment.

Here we show the initial steps in the recycling of hexaferrites. Material is recovered mostly from TV screens as a proof of concept. The recovered material is mashed until reduced to a size appropiate for milling. Then they are grinded, wet milled and finally ball milled and annealed in air. We have characterized the material at different stages of the process by vibrating sampel magnetometry, x-ray diffraction and Mössbauer spectroscopy. The characterization shows no significant degradation in the chemical composition. The resulting powder has been used for the production of the first batch of ferrite recycled injection molded magnets in a production line. Despite a slight decrease in the coercivity, attributed to the microstructure, the material seems to be competitive with the commercial grade one.

We gratefully acknowledge support from grants TED2021-130957B-C51 and TED2021-130957B-C54 funded by MCIN/AEI/10.13039/501100011033, and by the "European Union NextGenerationEU/PRTR" and the EIT Raw Materials funded INSPIRES project (20090).

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ABSTRACT

Mössbauer Studies of Ni-Mn-Sn Based Metamagnetic Shape Memory Alloys

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In recent years, Heusler-type Mn-rich Ni-Mn-X (X = In, Sb, and Sn) magnetic shape memory alloys have become the focus of intense research due to the observation of magnetic-field-induced martensitic transformation, MT [1-5]. In these alloys a metamagnetic phase transformation from a weak magnetic martensitic phase to a ferromagnetic austenite phase occurs, resulting in a large magnetization difference, ΔM, across MT. Associated with this behaviour, some interesting properties such as large magnetocaloric, giant magneto-strain and giant magnetoresistance effects have been reported, which make these alloys excellent candidates for multifunctional applications. With the objective of increasing the ΔM at the magnetic field induced MT, understanding the role of the composition, structure, microstructure and magnetic coupling in these alloys is essential.

The effect of combined mechanical and thermal treatments in the magnetostructural properties is studied in a Ni-Mn-Sn metamagnetic shape memory alloy, by 119Sn Mössbauer spectroscopy [6]. The influence of both the Co addition and the internal stress on the atomic level magnetism is comparatively studied in Ni50Mn37Sn13 and Ni45Mn38Sn13Co4 alloys by magnetic measurements and 119Sn Mössbauer spectroscopy. The results show that the saturation magnetization and the hyperfine field follow the same temperature trend. The internal stress state is investigated by subjecting the samples to milling and annealing treatments, and tracking the singlet component revealed by 119Sn Mössbauer spectroscopy [7].

In a previous work, Mn-based Mn-Ni-Sn doped with Fe alloys were investigated concluding that the Fe addition up to 4 % improves the mechanical properties and reduces both MT and its hysteresis resulting in an increment of ΔM [3]. 57Fe Mossbauer spectroscopy measurements were carried out in the 20 to 300 K temperature range on Ni42Mn40Sn10Fe8 powder alloy and Mn49Ni38Fe4Sn9 melt-spun ribbons. The results obtained are discussed in terms of different physical models.

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ABSTRACT

Mössbauer and XPS Studies of β-Hematin Crystals: Comparison of Two Synthesis Methods and Two Antimalarials

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It is now accepted that detailed knowledge of the physico-chemical characteristics of the β-hematin crystals, *i.e.,* the synthetic version of the natural hemozoin crystals, is important for the design of antimalarial medicines and for malarial diagnosis. In this work, we study the crystallographic, morphological, and Mössbauer spectral characteristics of β-hematin crystals, when these are prepared from hemin in aqueous and aqueous-oily mediums in the presence of two common antimalarial medicines: chloroquine and artesunate. The surface chemical states of the elements present in the materials were studied by X-Ray Photoelectron Spectroscopy (XPS). The room temperature Mössbauer spectra of all samples were fitted using the Blume-Tjon model, which assumes that the magnetic hyperfine field (H) fluctuates stochastically between +H and −H along the electric field gradient z axis with asymmetry parameter η = 0. The Mössbauer spectrum of the β-hematin micro-crystal synthesized in aqueous medium was adjusted by introducing two spectral components with low (*i.e.* more asymmetric) and high (*i.e.* more symmetric-like) average flip frequencies. In contrast, the spectrum of β-hematin synthesized in aqueous-oily medium was fitted with a single spectral component of low average flip frequency. The asymmetric spectral components are ascribed to relaxation phenomena of the iron ions located at the Fe^{3+}-PPIX units of the centrosymmetric dimmers. The more symmetric-like component suggested closer Fe^{3+} – Fe^{3+} distances in comparison to the more asymmetric one. The Mössbauer spectra of the micron sized crystals changed from asymmetric, for the samples prepared in the absence of antimalarias, to more symmetric-like, for those crystals prepared in the presence of the two antimalarials. Therefore, Mössbauer spectroscopy suggested that the spectral characteristics exhibited by the β-hematin crystals depended upon the reactive medium, and the type and the concentration of the antimalarial medicines. The XPS results suggested that the surface chemical state of iron at the surface is more complex than that initially expected. Besides the high spin Fe(III) species observed by Mössbauer spectroscopy, XPS revealed the additional presence of both high and low spin Fe(II) species. The concentration of these species appear also to depend on the reactive medium and the type and concentration of the antimalarial products. These species are not the result of sample degradation upon the X-ray irradiation during the XPS examination but rather genuine chemical species present in the surface of the samples.

ABSTRACT

Mössbauer Spectroscopy for Fe Electrodes in Fe-based Batteries and Single Atom Catalysts

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In some present problems related to devices or processes which involve metals which are scarce or difficult to recycle, iron can play an outstanding role. In particular, the search for substitution of Li batteries or catalysts containing nobel metals can be addressed with Fe. Mössbauer spectroscopy is a key technique which contributes to the determination of phases and microstructure of these types of materials. In the present talk I will show two cases. On one hand, the contribution of Mössbauer spectroscopy to the characterization of the microstructure of Fe electrodes for Fe-air batteries. On the other hand, the characterization of a single atoms catalyst (SAC) with Fe intended for proton-exchange membrane cell fuels (PEMFC).

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ABSTRACT

Inversion Degree Determined by Mössbauer Spectroscopy: The Case of $(Mn_{0.2}Co_{0.2}Ni_{0.2}Cu_{0.2}X_{0.2})Fe_2O_4$ (X=Fe, Mg) Spinel High-Entropy Oxides Prepared Via Reactive Flash Sintering

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We have achieved the successful synthesis and sintering of spinel-type $(Mn_{0.2}Co_{0.2}Ni_{0.2}Cu_{0.2}X_{0.2})Fe_2O_4$ (X=Fe, Mg) high entropy oxides through a single-step process utilizing Reactive Flash Sintering, RFS, technique. A single phase was obtained in just 30 min at a furnace temperature of 1173 K, identified with a spinel crystal structure $Fd\bar{3}m$. The structural and magnetic characteristic of the prepared compounds were assessed through various techniques, aiming to understand the correlations between functional properties and crystal structure. Mössbauer spectroscopy reveals that all specimens adopt the inverse spinel structure, in agreement with X-ray Photoelectron Spectroscopy results. Additionally, both techniques facilitated the determination of the valence state of Fe ions, confirming the presence of Fe^{2+} in the Mg-free compound.

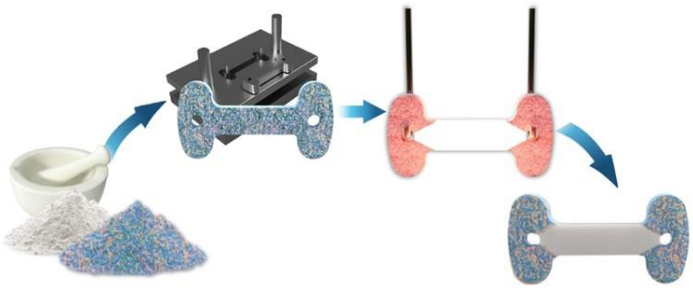

Figure 1: Schematics of RFS, in which the pressed dog-bone sample of the precursors powders is synthesized and sintered in a single step.

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ACKNOWLEDGEMENTS

VII PLAN PROPIO DE INVESTIGACIÓN Y TRANSFERENCIA

IBERMOSS2024 conference was funded by Vice-rectorate for Research of the University of Sevilla, VII Plan Propio de Investigación y Transferencia, VII-PPIT, *Convocatoria de Ayudas para la Organización de Proyectos Internacionales (Proyecto: 2024/00000306 - IBERMOSS 2024)*.

Participation of Emeritus CNRS Research Director Dr. Jean Marc Greneche was funded by Faculty of Physics the University of Sevilla under *Convocatoria de ayudas para actividades de divulgación y orientación de la Facultad de Física de la Universidad de Sevilla*.

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The use of the Grade Hall for the presentations was gently allowed and assisted by Faculty of Biology of the University of Sevilla.

Centro de Iniciativas Culturales de la Universidad de Sevilla

CICUS and Ancient Fund and Historical Archive of University of Sevilla are acknowledged for their gently assistance in social event.

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KEY NOTE CONFERENCES

MONDAY May 20th

^{57}Fe Mössbauer Spectrometry Applied to Solid State Chemistry and Physics

Dr. Jean M. Greneche

TUESDAY May 21st

Structural and Physical Properties of Fe Nanostructures

Dr. Jean M. Greneche

Nuclear Techniques for the Study of Matter. Positron Annihilation Spectroscopy and Neutron Scattering Techniques

Prof. Fernando Plazaola

INVITED SPEAKERS

Jean M. Greneche *Univ du Mans*

Fernando Plazaola *UPV/EHU*

Pere Bruna *Univ Pol Catalunya*

Benilde Costa *Univ Coimbra*

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