


Scientific Cooperation
between the Institute of Atomic Physics (IFA), Romania
and the Alternative Energies and Atomic Energy Commission (CEA), France

Joint Research Projects 2015

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Page

Table of Contents

Table of Contents

Page

Project C2-01

Development of innovative binders for the stabilization/ solidification of low- or intermediate-level radioactive wastes containing aluminium

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The project aims at investigating alternative cement systems to provide innovative solutions for the solidification and stabilization of problematic historic wastes containing aluminium metal. The objectives are threefold:

(i) compare the potential of calcium sulphoaluminate, magnesium phosphate, calcium phosphate and magnesium silicate binders to mitigate the release of hydrogen by a solidified waste form due to corrosion of aluminium,

(ii) assess the feasibility to reduce the corrosion of aluminium still further by the addition of a corrosion inhibitor in the mixing solution, and

(iii) develop, for the two most promising cementitious systems selected from steps *(i)* and *(ii)* a formulation checking the desired criteria for waste conditioning.

The project involved multi-disciplinary research groups with skills in materials science, physico-chemistry, analytical chemistry, and process engineering.

Step 1: Screening of binders (2012)

Pastes were prepared with cements from the four investigated systems (calcium sulfoaluminate, magnesium phosphate, calcium phosphate, and magnesium silicate), with or without aluminium. The phase assemblage of the hardened materials were characterized using X-ray diffraction and thermogravimetry. The pore solution of the material was extracted using pressure and analysed by ICP-AES, pH-metry and ionic chromatography.

The production rate of hydrogen by samples containing aluminium was investigated periodically sampled and analysed for its hydrogen content using gas chromatography. At the end of the trials, the transition zone between metal and cement paste was characterized using scanning electron microscopy, EDS analysis, and X-ray diffraction, the different cementitious systems were ranked as a function of their capability to mitigate aluminium corrosion.

Step 2: Investigation of corrosion inhibitors (2013)

A literature review of the corrosion inhibitors of aluminium was performed. The selected candidates were tested using different cementation systems. The corrosion rate of aluminium was measured as a function of the nature and concentration of the inhibitor added to the synthetic pore solution of the most promising binders identified in step 1 and binders were investigated. Then, the possible interactions between the most effective inhibitors and binders were investigated. Some cement pastes were prepared by spiking the mixing solution with the inhibitor. The influence of the inhibitor on the properties of the hardened material (strength, volume stability) was assessed. Finally, aluminium rods were encapsulated using the most promising cements and inhibitors, and the materials were characterized as previously described in step 1 (hydrogen production rate, solid phase assemblage, microstructure and composition of the transition zone between the binder and the aluminium rod).

Step 3: Design of a cement formulation checking the criteria for industrial application and final disposal (2014)

The aim of the third step of the project was designing a pumpable, flowable and self-levelling grout using the most promising binders and inhibitors. The material will also have to check the specifications for a final disposal of the waste packages in a surface repository. The investigated recipes were tested at the laboratory scale (volume of few litres) and optimized for their cement, water, and sand contents. The sand was carefully selected as a function of its composition and granulometry to get a good packing of the particles in the grout and improve its final strength. The elaborated materials were characterized using the conventional tools of civil engineering. Particular attention was paid to the heat output at early age, in order to keep an acceptable thermal excursion when the grout will be cast in large volume canisters. Experimental matrices were prepared and disposed in simulated (fast damaging) and normal repository (Romanian National Repository for Radioactive Waste Baita-Bihor) conditions.

Step 4: Experimental data correlation in order to validate the optimum matrix for conditioning low-or intermediate-level radioactive waste containing aluminium (2015)

In the last step there was made a detailed evaluation of the results obtained in order to choose and validate the optimum recipe for preparing the innovative materials for conditioning low-or intermediate-level radioactive waste containing aluminium. Also the aspects that require further investigation were pointed out, sketching new possible ways to approach them and opportunities for future collaboration.

Anticipated outcomes

The project will help finding a solution to treat problematic wastes containing aluminium. In this project it was possible to carry out a comprehensive study on a large number of alternative cementitious systems, and to build a database allowing a true comparison between the different types of binders. Besides, it was an opportunity to share its expertise on nuclear waste cementation. It will also improve the knowledge of alternative cement systems, and their interaction with electropositive metals. It should be recalled that, up to now, these wastes can't be encapsulated in a cement matrix and are temporarily stored in silos and ponds. From a scientific point of view, the project allowed a better understanding of inorganic binders which are much less known than ordinary Portland cement.

Cooperation perspectives

In the future, the cooperation between CEA and IFA initiated with aluminium-containing wastes might be extended to the conditioning of other types of wastes. IFIN-HH is interested in the conditioning of secondary wastes generated from inorganic sorbent and reverse osmosis liquid waste processes.

Project webpage address: http:// proiecte.nipne.ro/ifa-cea/1-proiecte.html

Project C2-02

Functional surfaces obtained by electroplating of nano-sized dispersed ceramic phases with metals for use in the nuclear industry to increase the corrosion and tribocorrosion resistance

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Project motivation

Conventional metals and alloys perform poorly in many applications where metal components are subjected to aggressive environments and mechanical friction conditions. In this project, it is proposed to fabricate nano structured metal matrix composite coatings using electrodeposition and co-deposition techniques of nanosized ceramic dispersed phases with metals. Significantly improved tribological and corrosion properties are desired from these nanostructured coatings.

Project objectives

- Making functional nano-structured surfaces by electrolytic deposition of nano-dispersed particles (mechanism and kinetic of co-deposition process of nanodispersed phases with metals).
- Obtaining of nanocomposite coatings on support materials at optimum parameters for performing the specific characterization tests in working environment.
- Characterisation of coating thicknesses, surface morphologies and composition by SEM-EDX, XRD, adhesion, roughness and micro hardness.
- Characterisation of corrosion, wear – corrosion (tribocorrosion) properties of nanocomposite coatings in specific environments.

Results

In Figure 1 there are presented as summary the results obtained in fabrication of functional surfaces by electro-codeposition process, kinetic and mechanism of nanoparticles codeposition with metallic matrix, changes in surface morphology by nanoparticles inclusion into nickel matrix, structural modification induced by nanoparticles as well as the increasing corrosion resistance by increasing the amount of nanoparticles into plating electrolyte. The

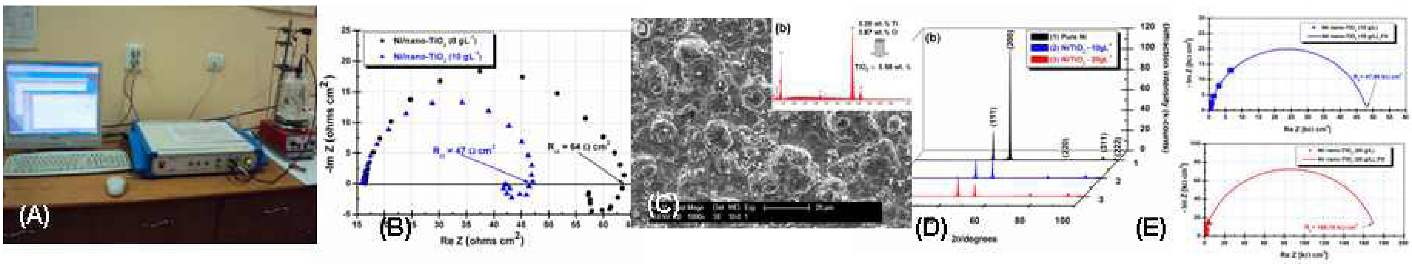

Fig.1. *Electrochemical fabrication of nanostructured composite coatings: (A) – Electrochemical equipment for electrodeposition and corrosion studies; (B) – Comparative Electrochemical Impedance Spectroscopy Diagrams (EIS) obtained for the kinetic and mechanism of nanoparticles codeposition with metallic matrix during electroplatiung process; (C) SEM – EDX surface characterization of nanocomposite coatings; (D) – X-Ray Diffraction patterns showing structural modification by nanoparticles inclusion and (E) – EIS diagrams showing the effect of nanoparticles concentration into electrolyte on corrosion resistance of nanocomposite coatings obtained.*

nanocomposite coatings showed good adherence to stainless steel support, a coating thickness from 10 to 100 micrometers and about 12 wt % of nano dispersed phase included into metal matrix with enhanced properties (nanohardness, corrosion and wear resistance in dry and wet conditions. In the tribocorrosion experiments it was used a solution that simulates at room temperature the corrosion environment from the primary circuit of PWRs in order to consider the real impact of the coatings obtained. Figure 2 shows a summary of special characterization of nanocomposite coatings in tribocorrosion system.

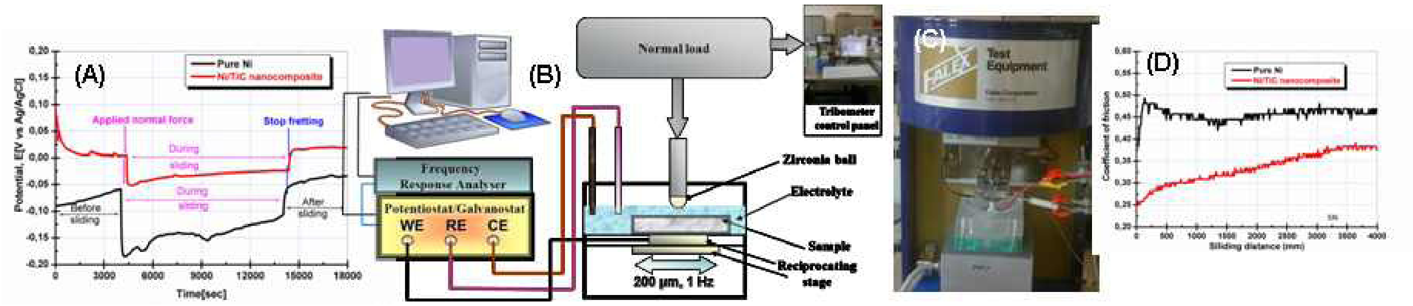

Fig. 2. *Characterization of nanocomposite coatings in tribocorrosion conditions: (A) – In –situ Electrochemical measurements showing the open circuit potential before, during fretting and after stopping the sliding with the better behaviour of nanocomposite coatings; (B) – Schematic set-up of tribocorrosion tests for simultaneously measures of electrochemical and mechanical parameters; (C) Tribometer to impose normal forces and measure the friction coefficients with electrolytic cell containing the tested sample (coating) and (D) – Specific friction coefficients diagrams.*

Dissemination of the results

In summary the results were disseminated by ISI and BDI publications, thesis, and presentation to international symposiums and conferences as follow:

Authors / Paper title / Journal
A. I. Pavlov, L. Benea, J.-P. Celis, L. Vazquez, *Influence of nano-TiO2 co-deposition on the morphology, microtopography and crystallinity of Ni/nano-TiO2 electrosynthesized nanocomposite coatings.* **Digest Journal of Nanomaterials and Biostructures**, Vol. 8, No. 3, July – Sept. **2013**, p. 1043 – 1050. **ISI / I.F.=1.092.**
L. Benea, A. I. Pavlov, *Ni-TiO2 nanocomposite coatings as cathode material for hydrogen evolution reaction*. **Optoelectronics and Advanced Materials – Rapid Comunications**, Vol. 7, No. 11 - 12, P 895-899, Noiembrie - Decembrie 2013, p. 895-899.**ISI / I.F.=0.402**
Lidia Benea, Eliza Danaila, Jean-Pierre Celis, *Influence of electro-co-deposition parameters on nano-TiO2 inclusion into nickel matrix and properties characterization of nanocomposite coatings obtained*. **Materials Science & Engineering A.** Vol 610, 29 July **2014**, p 106-115. **ISI / I.F.=2.108.** http://dx.doi.org/10.1016/j.msea.2014.05.028
Lidia BENEA, Sorin – Bogdan BAȘA, Eliza Dănăilă, Nadège CARON, Olivier RAQUET, Pierre PONTHIAUX, Jean-Pierre CELIS. *Fretting and wear behaviors of Ni/nano-WC composite coatings in dry and wet conditions*. In publication. Manuscript Number JMAD-D-14-02367 in **Materials and Design**. ISI / **I.F=2.913.**
Eliza Mardare, Lidia Benea, Iulian Bounegru, Electrochemical Modifications of Titanium And Titanium Alloys Surface For Biomedical Applications – A Review. The Annals of "Dunarea De Jos" University Of Galati, Fascicle IX. Metallurgy and Materials Science, N0. 1 – 2013, p. 68 – 78, ISSN 1453 – 083X. BDI.

12 paper presented oral/poster at international/national conferences.

1 PhD Thesis (Dr. ing. Adina Ionica PAVLOV), **1 Master Thesis** (Student – Roman Ortanța), **2 Bachelor Thesis** (Students Bașa Bogdan – Sorin, and Chiriac Andrei Mihai) coordinated by Prof. Dr. Lidia Benea.

Cooperation perspectives:

Taking into account the promising results both partners decided to propose another project in the next call in order to continue the collaboration. By continue collaboration it will be create the poll of excellence in the field of nanostructured functional surfaces obtained by electro-codeposition of nanosized ceramic dispersed phases with metals, corrosion – tribocorrosion by UDJG-CC - ITES (RO) and CEA Saclay – Ecole Centrale Paris (F). The international visibility of research teams from both countries will increase through publications and international conferences. The training of Human resources will improve the research qualities of young researchers and PhD students.

Project webpage address: http://www.nanosurfcorr.ugal.ro

Project C2-04

AMS analyses of concentrations of hydrogen isotopes and other elements in tiles dismounted from the Toroidal Pump Limiter at Tore Supra Tokamak

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In the course of the on-going project, performed between 2012 and 2014, our research was focused on the investigation of deuterium retention in different tiles dismounted from the toroidal pump limiter (TPL). Quantity determinations of the deuterium retention was performed by applying the Accelerator Mass Spectroscopy (AMS), upgraded to perform depth profiling of concentrations. Such complex and sensitive analyse are very important when the samples to be studied were exposed to different erosion factors and deposition areas. AMS, totally opposite but complementary to the classical techniques, is not detecting the radioactive agents by their emitted radiation (α, β, γ), but it selects and counts ion atoms individually, one by one. The method has the highest analyzing sensitivity known today, which is 10^{-15} (ratio: isotope/element). One of the AMS facilities of IFIN-HH Bucharest was upgraded and can perform depth profiling of concentrations. A dismounted sector of the Toroidal Pump Limiter (TPL) of Tore Supra (TS) was investigated by post mortem analyse of the deposition on the tiles. A relation of the enhanced deposited areas with the magnetic field geometry in the TS was clearly established. Raman microscopy measurements have shown that the carbon samples have an amorphous structure with anisotropic structure and the retention deuterium was following the anisotropy.

Project objectives - The main objective of the project is to investigate and measure the deuterium accumulation in the Toroidal Pump Limiter at Tore Supra. The distribution of particle retention on the limiter has also to be studied. Another important part of the project is dedicated to the elemental deposition studies on the protection tiles. By use of AMS and ICP-MS methods deposition and erosion areas on tiles were characterized concerning their elemental composition.

Obtained results - At this stage of the project AMS measurements have been performed to determine the retention and the depth concentration distribution of deuterium for a total of eleven (11) TPL samples removed from Tore Supra. The samples were cut from the original plates. For quantitative deuterium measurements standard samples and blank samples were used. All results were corrected for carter rim effects during the depth profiling of concentrations.

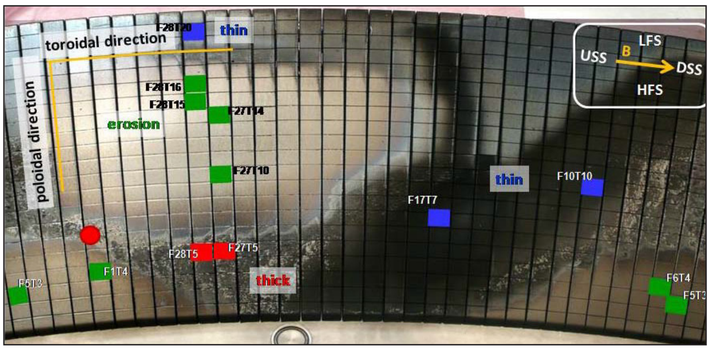

Fig. 1 *Part of the Toroidal Pump limiter of Tore Supra. Deposited or eroded areas are shown.*

TPL samples received from Tore Supra were further cut in several smaller slices. In this way it was possible to repeat the AMS analyse for the same location. It was noticed that repenting the measurement after six (6) month the deuterium content in the same surface layer has decreased. This trend was continuing and measurements performed after 1 year determined only very low deuterium concentrations. Fig. 2 presents the results of the in time repeated AMS analyses.

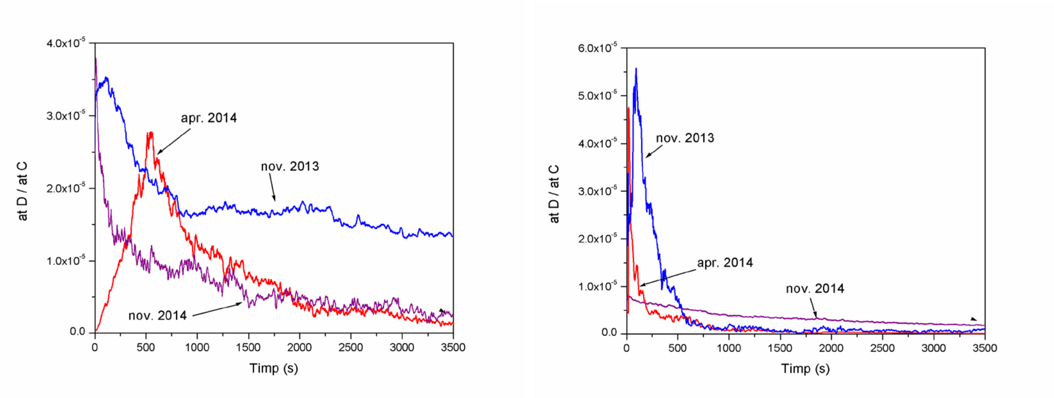

Fig. 2 *Evolution in time of the AMS depth profiles of deuterium retention in tiles of TPL. The spectra were measured at a time distance of six months.*

A special feature of the delayed AMS spectra is a maximum deuterium concentration located on the sample surface followed by a strong decrease towards inside of the tile. The diffusion of deuterium inside or outside the tile can be the reason for such a trend.

ICP-MS measurements revealed the elemental composition of the deposited or eroded areas.

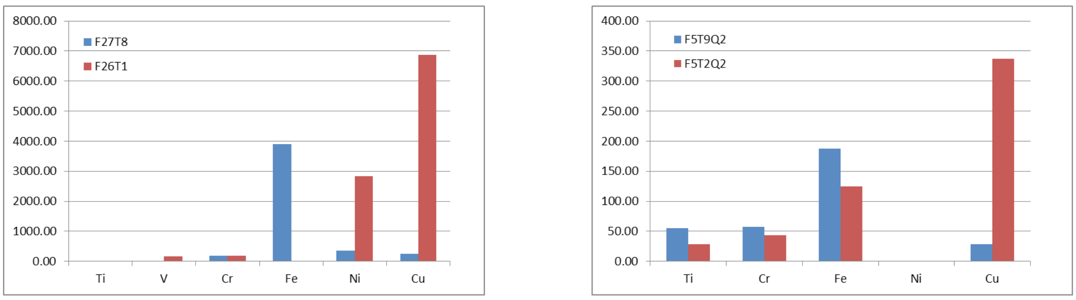

Fig. 3 *Results of ICP-MS analysis.*

Important quantities of Cu depositions are present. Fe, Ni, Cr, V were also determined. These materials were eroded from components and mechanical support and other components of TS and were transported in the region of the TLP.

Project webpage address: http://proiecte.nipne.ro/ifa-cea/2-proiecte.html

Project C2-05

Creation of national standards for some emerging pharmaceutical radionuclides to ensure the radioprotection of patients and medical staffs

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The main objectives of the research project IFA-CEA no. C2-05 (period 01.03.2012-28.02.2015) are:

1. Approach of the radionuclides of mutual interest, IFIN-HH/Radionuclide Metrology Laboratory (LMR), Romania – CEA/LIST/DM2I/LNHB, France, used for targeted therapy or diagnosis: ^{186}Re, ^{82}Sr-^{82}Rb, ^{177}Lu, ^{90}Y.
2. Common measurements to obtain primary standards of these radionuclides, by using absolute standardization methods; comparison of the results.
3. Delivery of an ampoule containing the same nuclide to the Bureau International des Poids et Mesures (BIPM) for participation to the International Reference System (SIR), in order to create or improve the international Key Comparison Reference Value.
4. Theoretical and experimental studies to improve or determine the nuclear decay scheme data of these nuclides, important for the dose calculations (half-life, photon emission intensity). Compilation and evaluation of the new data in the framework of the international co-operation "Decay Data Evaluation Project" (DDEP), and made available to the users through the NUCLEIDE database (*http://www.nucleide.org/DDEP_WG/DDEPdata.htm*).
5. Creation of secondary standards through the Ionization Chambers or gamma-ray spectrometry instruments which can be easily transferred to the nuclear medicine services in hospitals.
6. Dissemination of the results obtained, by common participation in international conferences/workshops and publishing articles in international ISI-quoted journals.

During the period 2012 – 2014, the following results were obtained:

- A new high-resolution X-ray spectrometer was assembled at IFIN-HH (new components purchased), turned on and tested; experimental calibrations in energy, energy resolution and detection efficiency were performed.
- The activity standardization and study of the radionuclides ^{177}Lu and ^{186}Re were performed at IFIN-HH/LMR as follows: purchase of the radioactive solutions from abroad suppliers; preparation of the radioactive solutions and sources, necessary for activity standardization; absolute activity standardization of the radioactive solution by the 4πβ(PC)-γ coincidence method, the variant of the detection efficiency extrapolation; gamma- and X-rays spectrometry measurements of the solid sources (check of the radionuclidic impurities, activity measurements; X-rays and gamma-rays emission intensity measurements); the calibration of the ionization chamber (IC) CENTRONIC IG12/20A for activity measurements, using vials with radioactive standard solutions; IC used for half-lives measurements with competitive uncertainties, below 0.1 %.
- IFIN-HH/LMR and CEA/LIST/DM2I/LNHB participated in an international comparison

(2013-2014): one vial with standardized solution of ^{177}Lu was prepared and sent to the BIPM, Sèvres, France, in the frame of the SIR system, key comparison BIPM RI(II)-K1. Lu-177; the results are under evaluation at BIPM.

- Common measurements were performed at LNHB, Saclay, France, for the activity standardization of ^{82}Sr-^{82}Rb and ^{90}Y. The new nuclear data determined at IFIN-HH were used to update the nuclear decay data evaluations of ^{177}Lu and ^{186}Re, by Dr. M.A. Kellett (LNHB).

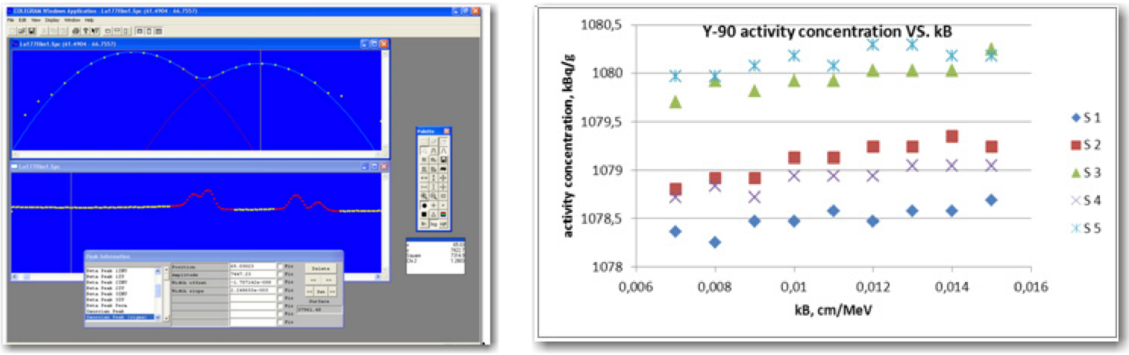

Fig. 1 *Experimental results: fitting the Hf KX-rays spectral region (^{177}Lu decay); ^{90}Y activity concentration measurements.*

- IFIN-HH organized the 5th international workshop of the DDEP co-operation (DDEP-2014), October 6-8, 2014, Bucharest-Magurele, Romania, *http://ddep14.nipne.ro*. The workshop was organized in cooperation with the International Atomic Energy Agency and DDEP, with the support of IFA, Romania and CEA, France. Five scientists from LNHB attended the workshop. Four papers about this project were presented in a special session.

Perspectives

The project IFA-CEA no. C2-05 will end in February 2015. A new Protocol of cooperation for 5 years will be signed by the two partners in 2015. New common research project proposals will be submitted in the future calls for projects.

References:

1. A. Luca, "*Nuclear Decay Data Evaluations at IFIN-HH, Romania*", Nuclear Data Sheets, vol. 120, 109-111, 2014;
2. A. Luca, M.-C. Lépy, "*Correction to the recommended γ-ray emission intensity of the 255.13 keV photons in the decay of ^{113}Sn*", Appl. Radiat. Isot., vol. 94, 147-148, 2014;
3. R. Ioan, M. Sahagia, A. Luca, A. Antohe, C. Ivan, "*Measurement of Lu-177 activity and its metrological traceability*", accepted paper – J. Radioanal. Nucl. Chem. (2015), DOI : 10.1007/s10967-015-4011-y;
4. M.A. Kellett, "*Evaluation of two emerging radio-pharmaceutic nuclei: ^{177}Lu and ^{186}Re*", and
5. A. Luca, M. Sahagia, M.-R. Ioan, A. Antohe, B.L. Neacsu, "*Experimental determination of some nuclear decay data in the decays of ^{177}Lu, ^{186}Re and ^{124}I*", papers accepted at the international conference ICRM-2015, www.icrm2015.at
6. PhD thesis, University of Bucharest, Romania: Beatris Luminita Neacsu, "*Contributions to the implement of gamma-ray spectrometry in some applications for nuclear medicine and radionuclide metrology*" (2012) and Mihail-Razvan Ioan, "*Study of the radiometrological and physical parameters of radiopharmaceuticals used in PET systems*" *(2014) (Scientific Coordinator, Dr. Maria Sahagia, IFIN-HH).*

Project webpage address: http://proiecte.nipne.ro/ifa-cea/3-projects.html

Project C2-06

Development and characterization of solid apatite matrices capable of storing inorganic pollutants: structure and adsorption processes

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It is well known that one of the major environmental problems is the global contamination with potentially toxic trace elements (PTTE). Lead has been widely used in the industrial field, for lead-based batteries, ammunition, paints and building materials. Due to their non-biodegradable behaviour and their incapacity of metabolization and decomposition, PTTE like Pb, Cu, Cd, Zn and Hg are the main contaminants of soils and ground or surface waters. Their progressive accumulation in the human body can cause significant health problems, inducing chronic illness which untreated, can lead to a painful death.

The project objective is to highlight the major contributions that could be made by engineered nanoparticles of hydroxyapatite and complex ceramic matrices for environmental applications such as soil and groundwater remediation. The project will test methods to obtain porous materials able to retain inorganic and organic pollutants in the laboratory, which will be subsequently tested in real contaminated sites.

The purpose is to develop scientific research on phosphocalcic apatites and to prepare a new porous material, capable of effectively eliminating inorganic pollutants like heavy metals from contaminated soils and waters.

The main goal of this project is establish a new method of synthesis in order to obtain solid apatitic matrices capable of retaining inorganic pollutants. Our studies will be focused on Pb^{2+} ions.

Hydroxyapatite (HAp) has a high sorption capacity for actinides and divalent metals. Furthermore, previous studies have revealed a high capacity for removing divalent ions from aqueous solutions and contaminated soils. For the removal of PTTE from polluted media by synthetic HAp different mechanisms have been reported, like ion exchange and substitution of Ca ions in HAp by metals ions. In order to improve the capacity of adsorption, it was shown that there are several factors that must be take into account, among them the type of divalent metal, the physico-chemical properties of HAp, the metal concentration, the solution pH, etc.

In accord with the reported work plan from the project proposal, the studies conducted until now have been focused on the development of complex ceramic matrices for environmental applications. Also, studies on Pb^{2+} ions adsorption by porous apatite and novel apatite complex materials from the aqueous medium have been conducted. The results reported during the first two years of this project were the synthesis and characterization of porous nano-hydroxyapatite bio-ceramics and new porous nano-composite material based on methyltrimethoxysilane coated hydroxyapatite.

The porous nano-hydroxyapatite bio-ceramics were synthesized using a sol-gel method. The gel was dried at 80° C for 96 h. The dried gels were individually heated at a rate of 50^0 C/min up to 600^0 C, 800^0 C and 1000^0C.

The methyltrimethoxysilane coated hydroxyapatite (MTHAp) was prepared using methyltrimethoxysilane and hydroxyapatite. The hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$) nanoparticles were prepared by setting the atomic ratio Ca/P at 1.67 and the methyltrimethoxysilane coated hydroxyapatite (MTHAp) was obtained by immobilizing hydroxyapatite (HAp) into a methyltrimethoxysilane foam. The removal performance of Pb^{2+} ions by the MTHAp composite powders was investigated by batch experiments, monitoring the change of Pb^{2+} ion concentration in the aqueous solution.

Various characterization techniques like: X-ray diffraction, Fourier transform infrared spectroscopy (FTIR), transmission electron microscopy (TEM), scanning electron microscopy (SEM) equipped with an energy dispersive X-ray spectrometer (EDS) and Raman Spectroscopy, were used to investigate the properties of the obtained novel porous materials. Also, the concentration of pollutants used in the batch experiments was determined by flame atomic absorption spectroscopy (AAS).The results showed that novel porous materials with controlled dimensions and properties were synthesized. The X-ray diffraction patterns confirmed that all the diffraction peaks could be assigned to the standard characteristic peaks of hexagonal hydroxyapatite and no secondary phases were detected, indicating that the phase of the samples was of pure HAp for all the investigated samples. In the case of methyltrimethoxysilane coated hydroxyapatite, the diffraction patterns support the fact that HAp nanoparticles were successfully coated with methyltrimethoxysilane without any structural changes. The impact of the lead concentration in the aqueous solution was studied by absorption experiments performed at 25°C. For this purpose MTHAp concentration in the range of 0.1– 0.9 $g \cdot L^{-1}$ at pH 5 was used. The measurements were performed on 500mL solution (pH 5) with an initial Pb^{2+} ion concentration of 63 $mg \cdot L^{-1}$. It was noticed that the removal efficiency increased proportionally with the Pb concentration. For Pb^{2+} concentration from 0.5 $g \cdot L^{-1}$ to 1.5 $g \cdot L^{-1}$, the removal efficiency was nearly 100%. In this case, the Pb^{2+} ions were completely removed from the solution.

The main objective of this project was to synthesize new porous nanocomposite materials based on hydroxyapatite and to investigate their ability to remove Pb^{2+} ions from aqueous solutions with a variety of initial Pb^{2+} ion concentrations and different pH values. In the case of MTHAp composite materials, the results obtained from the batch experiments showed that the powders exhibited the higher removal efficiency of Pb^{2+} ions at low pH. The lead removal capacity was the highest at pH 3 and the removal capacity of lead decreased slowly when the pH increased. After 24 h, most of the Pb^{2+} ions were eliminated from the aqueous solutions with various pH values and an initial Pb concentration of 563$mg \cdot L^{-1}$. This research showed that the MTHAp nanocomposite material is a promising adsorbent for Pb^{2+} ions from aqueous solution at various pH values and could be used as a purifier for wastewaters.

Project webpage address: http://infim.ro/projects/development-and-characterization-solid-apatite-matrices-capable-storing-inorganic

Project C2-07

Incorporated surface plasmons into core-shell fluorescent nanoparticles using microemulsion assisted photoreduction technique

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The principal object of this project was to obtain noble NPs (Au) combined with chromophores molecules that exhibit fluorescence enhancement by using microemulsion assisted photoreduction technique, which is highly flexible in synthesis and materials selection. The main objectives of the project were: i) design, synthesis and stabilization of Au NPs coupled with J-aggregated dyes that allow enhancement of fluorescence intensity; ii) optimization of synthesis of Au NPs coupled and combined with J-aggregates; iii) evaluation of optical properties of Au NPs coupled with J-aggregates.

The principal steps followed in achieving these objectives were:

i.1. Synthesis of metallic NPs in aqueous microemulsions. The AuNPs were synthesized in water in oil (W/O) micoremulsion system and they form a core which can support coating silica linked chromophores layer on their surface.

i.2. Synthesis of metallic NPs in non-aqueous microemulsions. In this case was used an oil in water (O/W) microemulsion system as chemical reactor for the synthesis of Au NPs – the Au NPs are stabilized at W/O interface forming a shell around organic phase which is able to support the encapsulation of chromophore molecules.

i.3. Study of factors controlling the size, structure and stability of NPs.

ii.1. Spectroscopic study of Au NPs combined with chromophores. The preferential location of the chromophore molecules among of oil-in-water interface, bulk oil and bulk water, in microemulsion, was determined by the spectral behaviour. For highlighting the solvatochromic effect and the preferential location of chromophore molecules in micelle and microemulsion systems was necessary to record absorption spectra of the molecular probe in stable media of colloidal systems of interest.

ii.2. Optimization of Au NPs shell thickness encapsulating chromophores for fluorescence signal enhancement. In this case modification of Au NPs shell thickness was obtained for different concentration of Au solution precursor. The fluorescence of chromophores molecule increased with increasing shell thickness due to decrease in non-radiative decay. The main scientific issue is to reduce Dexter quenching rate to a value below the radiative recombination rate, in this case by the positive (J aggregates like) coupling between fluorophores, which increases radiative recombination probably through superradiance.

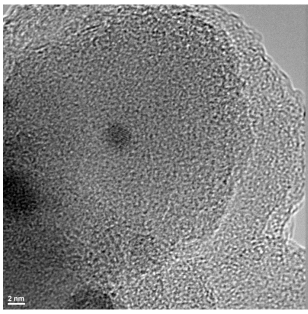
ii.3. Optimization of thickness of silica linked chromophores layer for fluorescence signal enhancement. In this case silica linked chromophores layer was modified in order to investigate the fluorescence signal enhancement. The results indicated an increase of the fluorescence signal of the chromophore molecules as the thickness of shell increased since chromophores moves further from the metal surface. ii.4. Testing RhB chromophore molecules for fluorescence intensity enhancement. Rhodamine B chromophore molecules were encapsulated into Au NPs to investigate the effect of overlap between the plasmon and absorption as well as emission peaks in the chromophores. In this case we obtained particles with dramatically increased brightness.

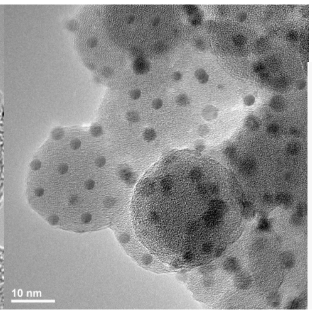
iii..1. Structural and morphological characterisation. The quantum size and environment effects were revealed by exploiting the absorption spectra of the Au NPs dispersed into liquid phase. The change in the fluorescence intensity in a close proximity of the probe molecules and the metal particles at the nanometer length scale, in solution, was evaluated by fluorescence studies.

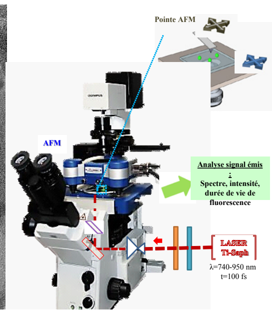
iii.2. Testing of optical properties of Au incorporated into core-shell fluorescent nanoparticles. Several studies were conducted in order to characterize the luminescence properties of hybrid metal-organic nanostructures. The goal was to evaluate the potential of plasmonic metal nano-antennas in promoting the molecular fluorescence.

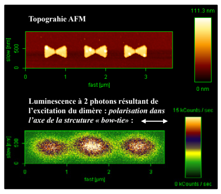
Expected results:

i) synthesis of Au nanoparticles coupled and combined with chromophore molecules;
ii) setting up the optimal conditions for synthesis of Au nanoparticles coupled and combined with chromophores for enhancement fluorescence intensity;
iii) optical characterization of Au nanoparticles coupled and combined with chromophores;
iiii) publication of two ISI articles [1, 2].









Scientific/technologic impact for promoting innovation and developments

We demonstrated in this project the possibility of using microemulsion assisted photoreduction technique for controlling both energy and electron transfer processes between the chromophore molecules and the gold nanoparticle with enhancing the fluorescence intensity. The demonstration of such bottom-up assembly processes by adapting soft nanotechnology techniques opened interesting perspectives in the whole domain of nanosciences and nanotechnology from biology (drug nanovectors) to optoelectronic devices. This comprises both the relative positioning of the fluorophores and their interaction with metallic nanoparticles associated with the characterization of luminescence properties, including its dynamics.

This NANOLIGHT project acted as a catalyst in promoting and developing existing cooperation between UPB and CEA by exploiting complementarities and creating synergies among current research fields of participating research teams, resulting in a mutual cross-fertilization of both human potential and infrastructure capabilities.

1. Nicoleta-Liliana OLTEANU, Aurelia MEGHEA, Marius ENACHESCU, Maria MIHALY, *Tunning gold nanoparticles size and surface charge by oil-in-water microemulsion template,* Digest Journal of Nanomaterials and Biostructures, Vol. 8, No. 4, 2013, p. 1687 – 1697.
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Project webpage address: http://cssnt-upb.ro/pro/ifa-cea-nanolight

Project C2-08

Actinides and Heavy Metals interactions on proteins and their biosensing

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2CEA Marcoule, Molecular recognition and interactions laboratory (LIRM), BP 17171 30207 Bagnols sur Cèze Cedex, France

The objective of the present project is to develop novel techniques for assessing biosensor surfaces built with self-assembled monolayers (SAMs) as well as the design of the biosensor and its testing.

Therefore, **the main tasks of the project** are: (i) reproducible characterization of a bio-functionalized surface in order to analyze the distribution of single proteins and evaluate their capacity of binding metals of interest. The main involved characterization techniques have been the scanning probe microscopy in imaging and spectroscopic modes providing information on the surface coverage and mechanical properties of the layer, its surface contact potential and dielectric constant variations, as well as confocal RAMAN microscopy for complementary characterization. (ii) designing and testing of the biosensor, mainly involving Electrochemical Impedance Spectroscopy (EIS) and related techniques.

The scientific methodology of this 3 year project included the following work packages (WP) and tasks:

WP.1. Developing self-assembled monolayers (SAM) on silicon and gold

Task 1.1. Identification of monoclonal antibodies/receptors that will bind to the heavy metals (LIRM)

Task 1.2. Identification of antibodies/receptors on SAM on silicon and gold (LIRM)

WP.2. Characterization of self-assembled monolayers before/after bonding with heavy metals

Task 2.1. Micro Raman characterization (UPB-CSSNT)

Task 2.2. AFM and SPFM studies, heavy metals (HM) interactions and electrical properties mapping (LIRM, UPB-CSSNT)

WP3. Biosensor design activities: Characterization of electrochemical behavior of SAM in a solution of heavy metals

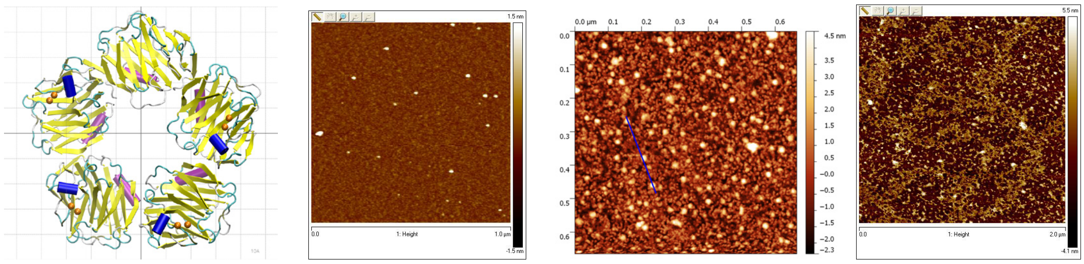
Task 3.1. Cyclic voltammetry studies on the SAM covered surfaces/electrode (CSSNT)

Task 3.2. Optimization of the electrochemical response of the electrode to the presence of heavy metals (UPB-CSSNT, LIRM)

As a result of the characterization of a list of proteins able to interact with actinides, C-reactive Protein (CRP) - which is a protein of 115kDa found in the blood where its levels rise in response to inflammation-.has been selected. Biochemical experiments confirmed the predicted binding site for UO_2^{2+} and it was demonstrated by surface plasmon resonance assays that UO_2^{2+} binding to CRP prevents the calcium-mediated binding of phosphorylcholine. Strikingly, the apparent affinity of UO_2^{2+} for native CRP was almost 100-fold higher than that of Ca^{2+}. Based on the above results, a novel screen-printed modified gold electrode through formation of a SAM involving mercapto-undecanoic acid (MUA), followed by the attachment of C-Reactive Protein (CRP), as a specific metal binding protein with a strong affinity to UO_2^{2+} ions, has been built and investigated.

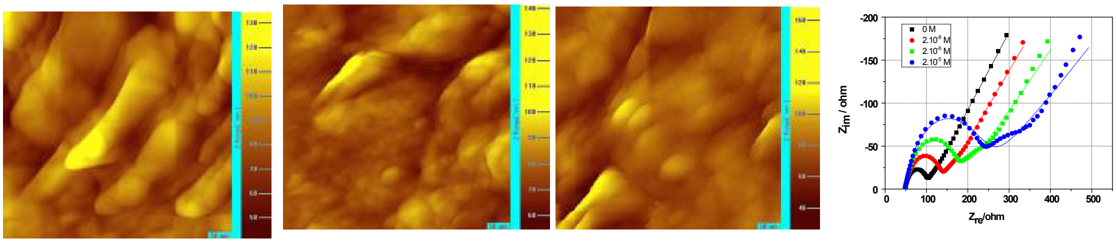
Comparative cyclic voltammograms of ferro/ferricyanide couple in buffer solution using bare Au, Au/MUA and Au/MUA/CRP electrodes showed the self-assembled monolayer formation and addition of stable thin protein layer. It was demonstrated that the Au/MUA/CRP electrode could be suitable for quantitative detection through EIS technique (Fig.2d) based on the proportional response of charge transfer resistance against uranyl ions concentration.

The performed research contributes to the development of monitoring capability of highly toxic components, including heavy metals ions. Both partners expressed their availability to valorize the obtained results towards development of other detection nano-tools based on metal-protein interactions.



(a) (b) (c) (d)

Fig.1 *– (a) Representation of the three-dimensional structure of human CRP; (b) AFM height image of 5 nM hCRP molecule deposited on mica; (c) Height image of chemically fixed antibodies on mica-grafted surfaces, with an expected height around 5 nm; (d) Topographic AFM image in air of a 100% COOH-SAM (self assembled monolayer) surface with antibody molecules deposited*



(a) (b) (c) (d)

Fig.2 *- AFM images of: (a) Au/SAM; (b) Au/SAM/CRP; (c) Au/SAM/CRP/*UO_2^{2+} *nanostructures (500 nm x 500 nm); (d) Nyquist plots of EIS recorded on Au/SAM/CRP electrode after 20 min. pre-concentration in solutions of various* UO_2^{2+} *concentrations*

Project webpage address: http://cssnt-upb.ro/993-2/ifa-cea-acheme/?lang=en

Study of new physics cases and preparation of SPIRAL2 experiments using the PARIS array

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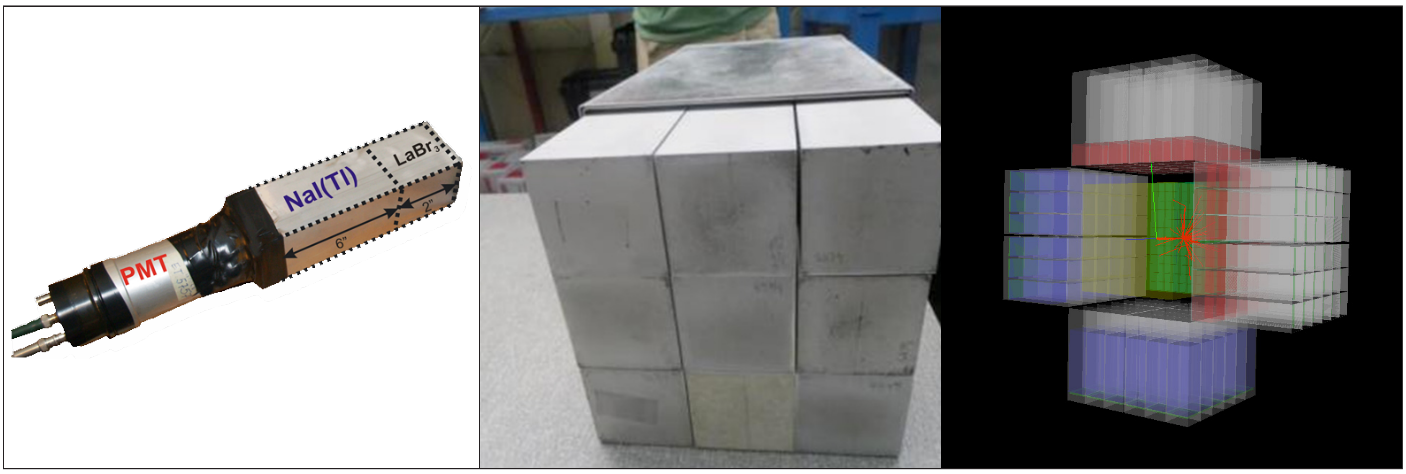
PARIS array is a multi-detector system under construction to be used in nuclear physics facilities with stable and radioactive beams for measurement of high energy γ rays. The basic component of is the phoswich scintillator detector shown in Figure 1a) and consisting in a 2”×2”×2” LaBr3:Ce crystal (BriLanCe380™) backed by 2”×2”×6” NaI:Tl crystal and coupled to a photomultiplier tube (PMT). The phoswichs are grouped (see Figure 1b)) in 3x3 modules that can be arranged in variable geometries to form around the target an inner shell providing timing, direction, gamma multiplicities and, together with the outer NaI shell, the energy. Due to the excellent characteristics of the new LaBr3:Ce scintillating material, as well the large detection volume and high granularity, PARIS will provide superior timing and efficiency parameters compared to other existing gamma multi-detectors and will complement the high energy resolution arrays based on Ge detectors.

PARIS is being developed in the framework of a large European collaboration built around the SPIRAL2 accelerator facility under installation at GANIL. The present project has given to the Romanian team the possibility to join this collaboration signing the Memorandum of Understanding and to contribute to development of the system, one the goal of the project. The other goal was to study and optimize several new types of experiments that can make use of PARIS array. These aims have been achieved through simulations using the GEANT4 framework of different PARIS configuration coupled to various detection systems and through participation in several test experiments performed by the collaboration at existing facilities. As planned, the simulated physics case were:



a) b) c)

Fig. 1 *a) Structure of a phoswich element; b) View of a PARIS cluster (3x3 phoswiches); c) PARIS-ACTAR TPC geometry* *i*) PARIS coupled to ACTAR TPC (Active Target and Time Projection Chamber, see Figure 1c)) for the study direct reactions induced (in inverse kinetics) by radioactive ions beams, *ii*) PARIS used in experiments with fast neutron beams available soon at NFS (Neutrons for Science) facility at SPIRAL2-GANIL and *iii*) PARIS in gamma beam experiments at ELI-NP facility under construction at IFIN-HH. The test of PARIS detectors and modules has been performed with beams provided by the Tandem accelerator at IPN-Orsay (France) and by the cyclotron accelerator at IFJ PAN in Krakow (Poland). These test experiments allowed to measure the energy and time resolution, linearity of calibration, efficiencies, response to high energy protons and to test the digital electronics system used data acquisition as well as the algorithms to be implemented for signal processing and for on-line/off-line data treatment up to gamma spectrum reconstruction. Regarding the digital signal processing, the time structure of gamma beams (100 macrobunches per second composed by 32 microbunches separated by only 16 nanoseconds) that will be available at ELI-NP imposed a study of the possibility to distinguish and analyses events separated by very short time interval. This possibility has been demonstrated in an experiment at CERN were an isomeric state of about 9 ns has been measured using a single plastic scintillator detector and fast digital acquisition.

During this project, the members of the team participated in a number of meetings and workshops organized by the PARIS collaboration and other scientific events:

- PARIS Collaboration meeting, TIFR Mumbai, India, Jan. 2013
- Workshop ELI-NP, Bucharest, Romania, Jun. 2013
- SPIRAL2Week, GANIL, France, Oct. 2013
- 'Physics with large arrays of novel scintillators', Dublin, Ireland, Jan. 2014
- AGATA Workshop, GANIL, France, Feb. 2014
- Workshop for PARIS experiments at IPN Orsay, IPN Orsay, France, Nov. 2014

The participation in PARIS collaboration will continue obviously well beyond the present project with common experimental campaigns and expected outcome will be mainly in the form of numerous publications, PhD and master's theses in nuclear physics and instrumentation. The first PARIS demonstrator is expected to be ready in 2015 and will consist of 5 clusters (45 phoswiches). One of the first experiments foreseen with PARIS will be in conjunction with AGATA multidetector for gamma rays and the VAMOS spectrometer at GANIL. These experiments will address several topics of interest, such as lifetime measurements in the A=18 region, SD bands at high spins in the Ca region, SD bands in the A=80 region.

Project webpage address: http://proiecte.nipne.ro/ifa-cea/4-proiecte.html

Project C3-03

Radiation effects in SiC: the role of the ratio between the grain size to displacement cascades length

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The main objective of this research project is to understand the role of the ratio of the grain size to damage cascade length on the effect of radiation damage. To achieve this goal, the Romanian team has the task to grow high quality SiC thin film samples. Advances in this area depend on the availability of good samples, having various degrees of crystallinity, high purity, good adhesion and low porosity that could be easily irradiated and then investigated to assess the effect of irradiation upon the physical and mechanical properties. The French team main task is to irradiate the samples and use various X-ray based techniques for their structural characterization. This project will synergistically combine the expertise of these two groups and investigate the effects of nuclear radiation on the crystalline structure, stoichiometry, and mechanical and optical properties of SiC thin films.

Project Stages

1. Fabrication of high quality-high purity SiC thin films by pulsed laser deposition and by magnetron sputtering;
2. Ion irradiation and post-irradiation characterization of SiC thin films obtained by PLD;
3. Fabrication and ion irradiation of epitaxial SiC thin films by PLD;
4. Post-irradiation characterization of epitaxial SiC thin films obtained by PLD;

Results

SiC thin films were grown by the pulsed laser deposition technique on Si substrates using high fluence and repetition rate under very low oxygen and water partial pressures at a substrate temperature of 1000 °C. The deposited films were dense, polycrystalline, with a low oxygen content in bulk and very hard. The crystalline structure of the films was investigated by X-ray diffraction (symmetrical and grazing incidence), while their mass density and surface roughness were obtained from simulations of acquired X-ray reflectivity (XRR) curves. The simulations indicated that the density of these films was between 3.15 to 3.20 g/cm3, very close to tabulated values for bulk SiC. This is an indication that the bombardment of the growing film by energetic atoms and ions during deposition resulted in a very dense structure. The chemical composition of the deposited films was investigated by X-ray phototelectron spectroscopy (XPS). The XPS survey spectra were acquired from the as-grown film and after 30 min Ar+ sputtering, when the contamination layer has been totally removed and the obtained chemical composition was therefore representative for the bulk of the deposited films. While the surface of the asdeposited film mainly consists of a SiO2-SiC mixture, the bulk was SiC with a very small amount of oxygen, around 1 at. %. Wear tests performed under ambient atmosphere at 900 °C showed that the deposited SiC exhibited the same friction coefficient and wear rate values as those measured at room temperature. After irradiation with Ar ion performed in France, the SiC thin film showed a decrease in hardness and Young modulus values. Raman investigations also evidenced changes in the chemical composition of the films. The studies that will be carried out during this project will contribute to a better understanding of the behavior of structure and mechanical properties of SiC under nuclear irradiation, opening new avenues for their technological applications. The end-product of the project is a contribution to a data base regarding the behaviour of nanocrystalline SiC thin films under intense radiation conditions. The acquired expertise will be very useful to deposit other carbides that are important nuclear materials. Such collaboration are the premise of a project development in the context of ERA or of future HORIZON2020 programs.

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Project website address: http://IFA-CEA-C3-03.inflpr.ro

Project C3-05

Education and training in the field of cultural heritage conservation by gamma irradiation

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The classical approach for biodecontamination of cultural heritage artefacts involves the individual treatment of each item, using common disinfectants and cleaning agents. This method implies mainly manual work, it is time consuming and cannot be applied for mass treatment of large quantities of artefacts. Fumigation with different poisoning chemicals is one of the methods that can be applied both for the treatment of individual items and for mass decontamination. Besides the microbiological risk, the use of classical decontamination methods adds the risk of using dangerous chemicals for the working personnel. Other methods such as deep freezing and anoxic atmosphere are environmentally friendlier but the treatment is not always effective against the resistant forms of the bio-contaminants: spores, eggs and pupae. Alternatively, gamma irradiation has major advantages: biocide effect guaranteed by reaching every part of the item, fast turnaround (hours), the highest degree of penetration (mass treatment), simplicity (the material is irradiated in its transportation or storage package), can be applied to composites and the treatment does not leave any toxic or radioactive residues.

The biocide effect of the ionizing radiation is indubitable. Radiation sterilization is currently one of the few existing industrial sterilization methods used in the field of the manufacturing of medical devices and pharmaceutical products with a market share of over 50% for the single use sterile medical devices. The efficiency and reliability of the method recommends it for the sanitization of cultural heritage items but its application is restrained due to the degradation effects encountered in the case of multiple and diverse natural polymers of the cultural items, already degraded by natural ageing or physical-chemical or biological factors.

This project intends to expand the results obtained in previous projects on the radiation treatment of wood, polychrome wood, paper, leather and textile cultural heritage artefacts by using advanced characterization techniques, organization of training courses, exchange of experience and knowledge transfer. After the irradiation treatment, both bioburden (microbial load of the items) and degradation effects (chain scission, oxidation, *etc.*) depend on the absorbed dose. High radiation doses will highly decrease the bioburden (exponential decay) and low doses will produce less degradation on the irradiated materials. Also, the use of radiation-curing resins to enhance the material properties is another very interesting application of gamma ray irradiation processes for cultural heritage artefacts conservation. It has been demonstrated to be very helpful for preserving hugely degraded wooden artefacts when classical consolidation was not sufficient, or to preserve the function of an artefact subjected to mechanical constraint.

The project has two main objectives:

1. Research of the radiation induced effects on cultural heritage items that will lead to a maximum allowed radiation dose for the treatment. The tests will be performed on both recent and naturally and/or artificially aged materials. For subtracting the cumulative effects, tests will be performed on constituents, raw materials and additives. The process specification for irradiation treatment will be built as first stage of the preparation of technological transfer of the use of the irradiation technology for cultural heritage preservation.

2. Knowledge transfer and knowledge dissemination through the organization of an annual workshop and participation to scientific meetings. The training component addresses important topics for the workers in cultural heritage deposits and laboratories: environmental safety of the workplace and means of reducing of the biological threats in working with highly bio-contaminated items and radiation effects on irradiated materials. These topics are dedicated especially to conservators and restaurators with the goal of clarifying misunderstandings related to the irradiation process and for increasing their capability for taking decisions on the appropriateness of application of the radiation treatment for a certain category of cultural heritage items. The transfer of the consolidation procedure of very degraded heritage wood artefacts by impregnation with resin

and radiopolymerisation from ARC-Nucléart, CEA-Grenoble to Radiation Processing Center of Horia Hulubei National Institute for Physics and Nuclear Engineering (IFIN-HH) is also envisaged.

Project's phases

1/2013. Studies on the requirements and limitations of irradiation treatment of cultural heritage artefacts. The transfer of the procedure of heritage wood consolidation by irradiation from ARC-Nucléart to IFIN-HH

2/2014. Advanced characterization tests pre and post irradiation of experimental models of cultural heritage artefacts

3/2015. Physical chemical tests before and after radiation treatment of cultural heritage artefacts

4/2016. Dissemination of project results

The project goals are aligned with the need of specialists in conservation/restoration of cultural heritage artefacts for new decontamination and/or consolidation methods. The synergy of the two laboratories to design and finalize the guidelines in using irradiation for the preservation of cultural heritage artefacts, in direction of the end-users, *i.e.* museum curators, conservators and staff in cultural administrations has to be emphasized. This guidelines work, in cooperation with other countries in Europe, is on progress with the support or the IAEA, Vienna, Austria, through Technical Projects and Coordinated Research Projects managed by different departments of IAEA. The two laboratories are actually the leaders in Europe and worldwide for this cultural heritage application of irradiation, and the only who can enhance the future development of this process, as well as the worldwide diffusion of the technique.

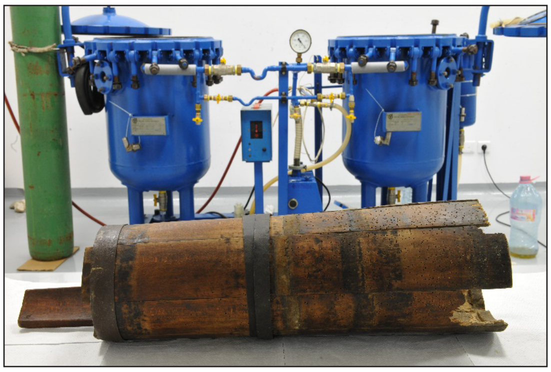

Fig.1. *Wooden churn before (left) and after (right) the consolidation; enhanced mechanical strength supports the standing churn own weight*

In 2014, with the help of experts from ARC-Nucléart, Grenoble, for the first time in Romania, the consolidation of a very degraded ethnographic wood artefact from the Viticulture and Pomiculture Museum of Golești, Arges county, was performed by impregnation with resin and radiopolymerization (Fig.1).

In 2013 and 2014 the First and the Second International Workshop of Nuclear Techniques for Study and Preservation of Cultural Heritage organized at the Horia Hulubei National Institute for Physics and Nuclear Engineering (**IFIN-HH**) were attended by numerous specialists from the cultural heritage field. The possibility of creation at **IFIN-HH** of a Virtual National/Regional Center for the Study and Preservation of Heritage was discussed. Also, in the frame of the project every year, a course on Irradiation techniques for cultural heritage is delivered to the students of the Master of Management of the Restoration and Valorization of Cultural Heritage of University of Bucharest.

By dissemination in scientific/professional meetings from cultural heritage preservation field it is expected that museums or owners of cultural heritage collections to become interested in the radiation treatment. Through collaboration, organization of workshops and communication of the results it is expected that the project team will definitely further develop its international cooperation capabilities by applying for funding in the program HORIZON 2020.

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Project webpage address: http://proiecte.nipne.ro/ifa-cea/7-proiecte.html

Project C4-01

Characterization of Superconducting Cables by X-ray Micro-Tomography

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Project general objectives

- Demonstrate that X-ray micro-tomography (μCT) can be used as a very efficient and reliable tool for retrieving the necessary information for constructing detailed 3D models of the Cable-in-Conduit Conductors (CICC).
- Upgrade the multi-physics models taking into consideration the experimental results from μCT examinations and validate their new configuration through confrontation with experimental tests campaigns.

Current reporting phase objectives

- Development of a combined 2D/3D transmission tomographic system optimized for geometrical measurement of superconductor cables internal structures with sub-voxel precision.
- Definition of the critical parameters to be examined and technologically assessed.

Estimated results

We believe that the previous experience of INFLPR in the design and fabrication of tomographic systems, in the development of advanced algorithms for 2D/3D reconstruction, and in the complex modelling of CICC structures is an essential asset for the accomplishment of these objectives at a high scientific level and in due time. The same applies for CEA experience on CICC design expertise for fusion technology, using synergies between elaborated codes relying on different physics domains.

Optimization of the μXCT setup

X-ray microtomography analyses of the typical CICC samples pose serious challenge due to their bulky shape (cylinders or rectangular cross sections of up to 30 mm characteristic dimension) and high effective atomic number (main elements Cu, Nb, Fe and Ti). For quantitative processing, one needs to reconstruct the strand diameter (≈0.8 mm) on at least 20 image pixels. High-resolution images (pixel size ≤ 40 μm) can be obtained only with a combination of high energy; high intensity and small focus spot X-ray source and high resolution / efficiency detector array.

We have tested two possible μXCT scanning methods: i) cone beam configuration associated with a high resolution flat panel detector and ii) fan beam configuration with line sensor based on individual scintillators. The cone beam configuration has the best X-ray beam utilization, but for high density and bulky samples, the images have a noticeable blur, mainly due to very intense X-ray scattering.

In the fan beam configuration, the intensity of the scattered X-ray can be reduced to around 5% of that encountered in cone beam scanning. In addition, the fan beam can make use of high detection efficiency of individual scintillators arranged in a line sensor. Our upgraded cone beam setup has been recently described [1, 2]. Figure 1 shows a view of the INFLPR 2D μXCT arrangement.

Qualitative analysis of CICC by μXCT

Once the CT configuration is selected and the scanning parameters are optimized (high voltage 210-215 kVp @ target power 20-25 W, time per slice <2 min) one proceeds to complete the visual inspection of up to 300 mm long reconstructed CICC samples. Stacks of fans projections (sinograms) with lateral resolution of ≈40 μm and axial pitch between 0.25 and 1 mm are thus recorded. Adequate software, (for example VGStudio MAX 2.x http://www.volumegraphics.com) is used to navigate inside the 3D rendered volume. For brevity, one illustrates the important qualitative analysis feature with the montage shown in Figure 2. The wrapping foil (0.1 mm) complex geometry and structural integrity is clearly revealed. All 486 strands are visible and their trajectories can be virtually followed. The cable pattern, the twist pitch sequence as well strands and voids distribution and the inter-strand and strand to jacket contact regions are identifiable.

The space resolution achieved with this technique can provide information about structural integrity (i.e. partially thinned by abrasion or even broken strands) after the service period of CICC.

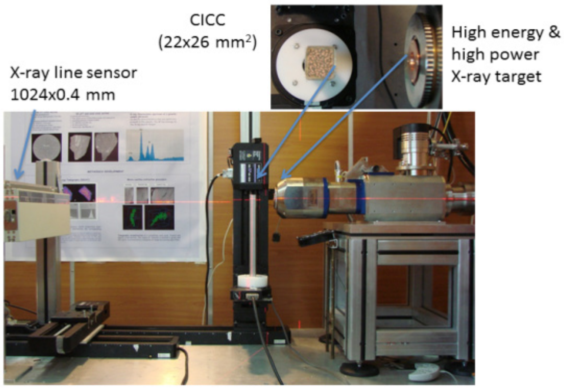

Fig.1 *View of the INFLPR 2D μXCT.*

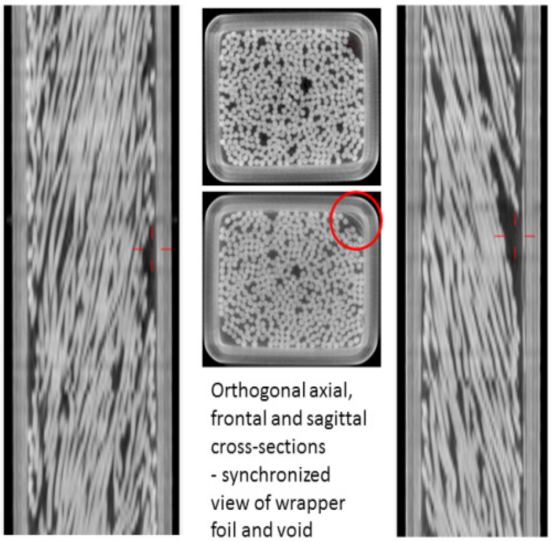

Fig. 2 *All constitutive elements of CICC can be noninvasively inspected. Corner void and wrapper foil overlapping are outlined.*

References

[1] I. Tiseanu et al., Fusion Engineering and Design, 82, (2007) pp. 1447–1453.
[2] I. Tiseanu et al. Fusion Engineering and Design, 86, Issue 9-11, (2011), pp 1646-1651

Project webpage address: http://tomography.inflpr.ro

Project C4-02

Development of the traceability of the calibrations in radiotherapy, radiation protection and medical imaging in Romania

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French project leader: **Dr. Dorin DUSCIAC**
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This project is, according to the statement "Development of the traceability of the calibrations in radiotherapy, radiation protection and medical imaging in Romania", a result of the cooperation between IFIN-HH and LNHB implemented in 2012-2013 (Joint Support Actions cooperation AS 1-02/01.10.2012). The initial Joint Support Action cited here above was approved in the frame of the IFA-CEA Agreement. The project is aimed at developing the traceability of the calibration for radiological protection, radiotherapy and medical imaging in Romania.

At present, in Romania, the medical imaging and the radiotherapy techniques are used on an increasingly larger scale. The patient safety during these radiological procedures depends on the accuracy of the measurements regarding the related radiological quantities. In order to assure the necessary accuracy of these measurements, a correct calibration of the measuring instruments is needed. The traceability of the standards and measurements is the way to an accurate measurement.

The aim of this project is to lead to an improved traceability for the measurement of the radiological quantities dedicated to radiological protection, medical radiological imaging and cancer radiotherapy; in the same time, the results of the research work done in this project will be useful for the dosimetry (radiological protection, but also beam dosimetry) for the ELI-NP Project.

This improved traceability will be implemented in several steps:

- The calibration of the Romanian standards in France, at LNHB (CEA),
- The development of a national primary standard in Romania, at CMRID (IFIN-HH),
- The extension of the range of validity of the existing national standard in France,
- The comparison of the national references of both countries once the national reference of Romania will be established.

This project will lead to an increased accuracy of calibrations and measurements in the field of radio diagnostics, radiotherapy and radioprotection in Romania. The achievement of the project will lead to better performances of the Romanian laboratory; this will be a strong basis for the participation of the Romanian laboratory (CMRID) in the R&D projects from the European R&D programmes in the field (e.g. EMPIR - European Metrology Programme for Innovation Research, EURATOM, Horizon 2020).

The project has two goals. The first is to develop the traceability of the national reference standard of Romania in high energy photon fields for radiotherapy and radiation protection of patients and workers, both for medical appliances of the ionising radiation and for new research facilities (i.e. ELI-NP). The first step will be to study at what extend the standard instruments of IFIN-HH can be calibrated for high energy photons and for other types of radiation (electrons, e.g.) at LNHB – French national calibration laboratory for the ionising radiation.

After establishing the possibilities and the conditions that are necessary in order to extend the traceability of the Romanian standards, the calibration performed at LNHB will allow for the development of a new calibration facility at IFIN-HH, in accordance with the new challenges in this field (for ELI-NP for instance).

In the second step, the LNHB helps the IFIN-HH to develop its own national reference. Within this frame, the LNHB will help the IFIN-HH to manufacture, test, and implement a primary standard dosimeter (cavity ionisation chamber) for the measurement of air kerma. During this study the LNHB will extend the range of validity of its references for the low dose rates. Once the Romanian national reference value will be established, the LNHB and the IFIN-HH will undergo a bilateral comparison. The result of this comparison will allow the IFIN-HH to validate its competences within the framework of the programs of international quality assurance.

In the first stage of this project, the design of a cavity – ionization chamber was achieved. This project is based on the experience and expertise existing at LNHB in field of the primary standard for dosimetry. The design took into account the important aspect that a cavity – ionization chamber must be used a primary standard, for absolute measurement of the air - kerma. Due to this aspect, a special attention was played the following:

- The selection of the material for the chamber wall, in order to assure the air-equivalence ;
- The selection of the material for the insulations, in order to have the lowest leakage current; the leakage current determines the lower limit of the measuring range of the chamber (Fig.1).
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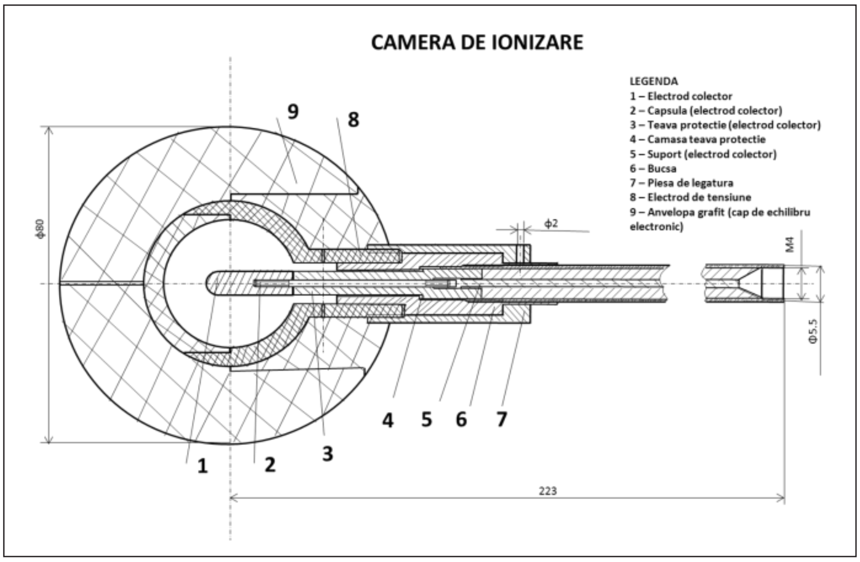

Fig. 1

The dimensions and the value of the sensitive volume of the chamber are also very important for an absolute method of measurement. In order to have a highest precision of the measurement, before connecting-up the chamber, the value of the sensitive volume shall be measured and certified by LNE-France.

Based also on the results of this project, IFIN-HH fulfilled the necessary requirements in order to be accepted as partner in the JRP project 14 RPT 04 Absorb (Absorbed dose in water and air) ; this project is coordinated by CEA (Project coordinator, Dr. Jean-Marc Bordy) and will start on 01 June 2015. The 14 RPT 04 Absorbed project was accepted in the European Metrology Programme for Innovation and Research (EMPIR). This Programme was has been approved by European Parliament and Council.

Project C4-04

Pyroelectricity in PZT thin films and multilayers

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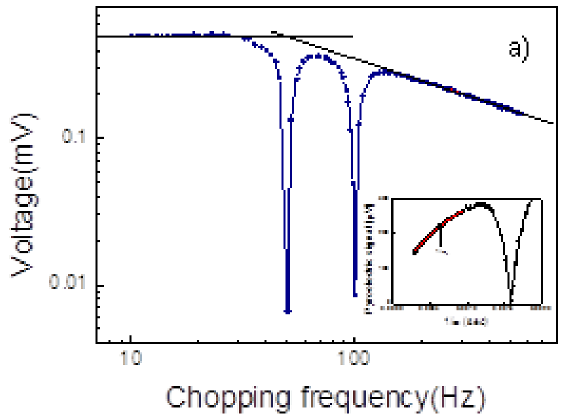
The main objective of the project is to study the pyroelectric properties in textured and epitaxial ferroelectric thin films and multilayers. The project is based on the concept of enhanced polarization in epitaxial layers due to strain imposed by the substrate, leading also to internal electric fields that make un-necessary the poling procedure used for ceramics. Basically PZT materials will be considered but lead free materials will be also taken into consideration as a possible alternative. The primary goal is to develop high sensitivity active elements for manufacturing pyroelectric detectors directly integrated on Si wafers. A secondary goal is to compare the pyroelectric properties in high quality epitaxial films grown by PLD on single crystal substrates with the pyroelectric properties of the textured films grown by low cost methods on buffered Si or Pt/Si substrates.

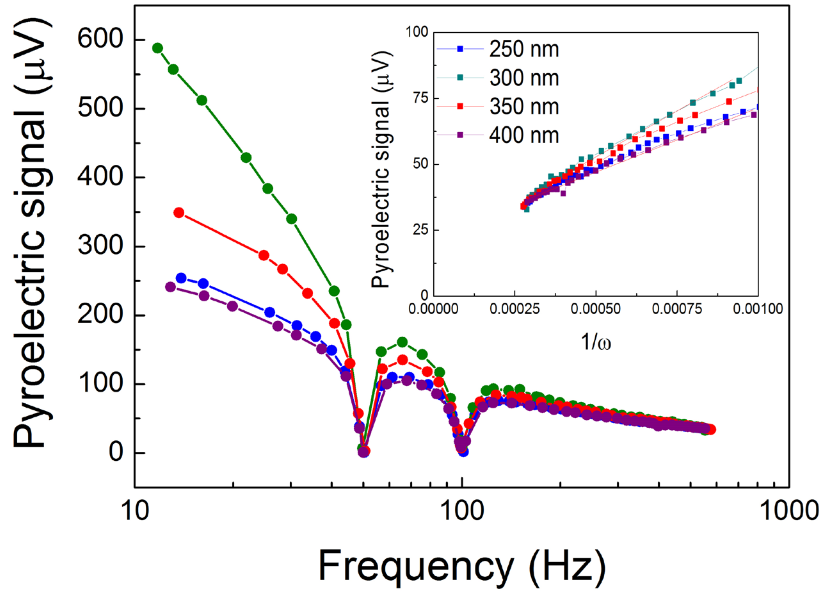
Among the issues that will be investigated during the project are: 1) Direct measurement of pyroelectric signal in high quality epitaxial films grown on single crystal STO substrates, by using the voltage operating mode, and correlation with the internal electric field and leakage current. The effect of the Schottky type contacts on the pyroelectric properties will be studied; 2) The development of pyroelectric active elements directly on Si substrates by using buffer layers as template for textured and epitaxial growth of ferroelectric films. This opens the way for a rapid integration of pyroelectric sensors with Si based electronic for signal processing. The growth of ferroelectric layers directly on Si will allow fabrication of membrane-like active elements (suspended detector) by etching the Si substrate. It is expected that such active structures will have larger temperature variations, thus larger pyroelectric signals; 3) Development of PZT multilayers by changing the Zr/Ti ratio. This offer the possibility to study two problems: (i) the effect of the concentration gradient obtained by varying the Zr/Ti ratio on the magnitude of the pyroelectric coefficient; (ii) the effect of interfaces on the figure of merit represented by the ratio between the pyroelectric coefficient and the dielectric constant; 4) Development of novel multilayer structures by combining PZT with polar semiconductors as ZnO (doped ZnO can also be used as an electrode); 5) Investigation of pyroelectric properties in some lead free materials. Barium-strontium titanate (BST) will be tested, if the time will allow preparing good targets in the 24 months time frame.

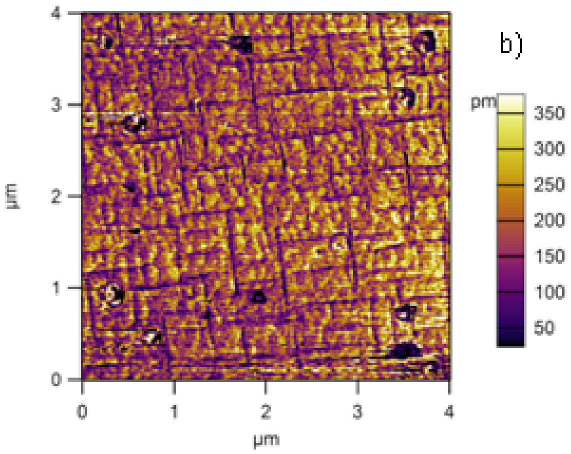
The project has innovative potential, especially regarding the multilayer structures which were not yet tested for pyroelectric detection. For example, a concentration gradient can produce a gradient in the spontaneous polarization and dielectric constant. Theoretical predictions claim that such gradient can lead to considerably enhanced values for the figure of merit but these were not tested experimentally on thin films of good structural quality.

The project activities are grouped in 3 main stages: 2014-growth of PZT thin films (by PLD at NIMP and by sol-gel at CEA); 2015-investigation of electric and pyroelectric properties; growth of new multilayer structures; comparison of pyroelectric properties; 2016-investigation of pyroelectric properties in suspended PZT layers; analysis of pyroelectric properties in lead free materials.

The two teams have a long collaboration record: a common IFA-CEA project for the period 2010-2013; a common proposal for Horizon 2020 (call H2020-ICT-2014-1; Title: Integrated – Smart Optical Sensors for Mobile AiR Testing; unfortunately did not passed the thresholds); a M-ERA net project that was selected for funding (call 2013); several common publications (most recent was accepted for publication in Journal of Materials Science). Therefore, there are very good premises that the collaboration will continue after the ending of the present IFA-CEA project under the form of common project proposals in the frame of various funding opportunities. Some important results were already obtained in the frame of the present project. The most interesting is that the pyroelectric signal can be amplified, at low modulation frequencies, by continuous illumination of the active pyroelectric element (epitaxial PZT film) with UV light. This was explained by the additive contribution of the photogenerated carriers to the pyroelectric current. The finding was published in Applied Physics Letters (see Ref. 1).







Left-a) frequency dependence of the pyroelectric signal obtained from an epitaxial PZT thin film; b)-the domain structure of the pyroelectric fim.

Above-UV amplification of the pyroelectric signal

1. L. Pintilie, M. Botea, and A. Iuga, "*Enhancement of pyroelectric signal by continuous ultraviolet illumination of epitaxial, Pb(Zr0.2Ti0.8)O3 films*", Applied Physics Letters **105**, 132901 (2014).

Project webpage address: http://www.infim.ro/projects/pyroelectricity-pzt-thin-films-and-multilayers

Project C4-05

New bioceramic nanocomposites with antimicrobial activity for biomedical applications

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The main purpose of the project is to develop new antibacterial materials for various applications. Also, the management informational system of the project will offer a share point infrastructure mapped on the scientific experiments data patterns and also mapped on specific environmental data acquisition protocols requested and recommended by the European Union.

This project is based on a multidisciplinary integrated approach involving physics, chemistry, microbiology, toxicology and medicine.

The novelty element of this project is represented by the use like antimicrobial agent of silver/zinc doped hydroxyapatite at nanometric scale. On the other hand, we propose to use for the first time the silver/zinc doped hydroxyapatite/collagen composite, to place on different types of wound. The ultimate goal of this project will be to obtain a new wound dressing used in different medical applications.

In support of accomplishing the general objective, the specific objects of the project are the following:

- obtaining and characterizing of the biomaterials based on silver/zinc doped hydroxyapatites and silver/zinc doped hydroxyapatite/collagen composite;
- in vitro and in vivo testing of the obtained biomaterials and composites;
- publishing the reference results in literature;
- participation at national and international congresses and conferences;
- patenting of the final product obtained.

The work plan is based on 3 main activity presented below:

A.1. Preliminary bibliography actualization, new experimental configuration. Development and characterisation of silver doped hydroxyapatite.Romania-France travel for scientific exchanges and experiments.

A.2. Development and characterisation of zinc doped hydroxyapatite. Romania-France travel for scientific exchanges and experiments.

A.3. Development and characterisation of zinc/silver doped hydroxyapatite in collagen matrix.

Both the French and the Romanian partners have well-established collaborations with research laboratories in the European Union and with industrial partners in the field of biotechnology and in the domain of biomedical applications.

The project will test methods to synthesis silver/zinc doped hydroxyapatite and silver/zinc doped hydroxyapatite/collagen composite able to inhibit the bacterial activity of different species of bacteria in the laboratory that will be implemented in real infected sites. The project's originality consists of the elaboration of the complex treatment protocol of acute and chronic cutaneous wounds using ceramic biomaterials at nanometric scale. The project is complex, necessitating the involvement of a multidiscipline partnership (medicine, biotechnology, biology, chemistry and physics) and collaborations such as the one proposed here with the CEA for the biological part. It will contribute in creating groundwork for the clinical studies regarding the use of composite biomaterials and in elaborating a treatment protocol for acute and chronic cutaneous defects.

The project objectives are to highlight the major contributions that could be made by nanoengineered particles of hydroxyapatites for biomedical applications such as inhibition and prevention of bacterial infections.

The formation of a new research staff specialized in new methods and techniques will create the opportunity for approaching new research directions in the priority domain of biotechnology. As a result NIMP will perform high quality and competitive research services and it could diversify the competence fields, bringing a real contribution to the attaining of the specific objectives of priority domain of biotechnology.

The project research favours the superior capitalization of low-priced and easy to obtain raw materials. From the technological point of view the results of the project envisage the permanent and dynamic need of producers of biomedical devices to adapt to the ampleness and rapidity of changes on the global market:

- Increase of quality and efficiency of products;
- Increase of safety and sustainability of operation by ensuring the level of functional performance parameters stipulated by European standards for similar products.

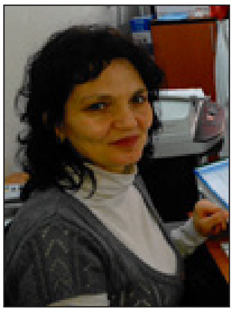
The most important economic impact of this project will be the general decrease of the expenses for medical assistance for patients with major tissue injuries.

The results of this project can form the basis of a greater cooperation in international programs such as FP7 and FP8 but also under contracts that concern the use of bioceramic nanocomposites with antimicrobial properties in biomedical applications. During the project other related topics of common interest can also appear in national and international programs. The cooperation between the 2 laboratories during this project will be the ground state for working towards developing a project in the newest and biggest EU Research and Innovation programme ever (HORIZON 2020).

Project webpage address: http://www.infim.ro/projects/new-bioceramic-nanocomposites-antimicrobial-activity-biomedical-applications

Project C4-06

Numerical Simulations for Radioactive Waste Disposal 2 – NSRAWD 2



Romanian project leader: **Dr. Crina Bucur**
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French project leader: **Dr. Alain Genty**
Laboratory for Applications in Thermal hydraulics and Fluid mechanics (LATF), CEA Saclay, 91191 Gif-sur-Yvette - France

To evaluate the safety of radioactive waste disposal in a near surface facility, it is essential to have a good prediction of radionuclides release from the disposal area and their transport through the geosphere and biosphere.

The NSRAWD 2 project proposes to continue and strengthen the ICN and CEA collaboration in the field of numerical simulation for radioactive waste disposal, combining experimental activities with computational work in order to improve and validate flow and transport numerical models and to determine important radionuclides transport properties such as dispersivity and retardation. The influence of cement barriers used in repository construction on radionuclide sorption is also considered in the project. Numerical models appropriate for each element of the disposal system already coupled in the Alliances Platform will be further improved in order to reduce the uncertainties and to get a more realistic approach for the performance and safety assessments of the Saligny LIL waste repository.

The objective of the project is to reduce the uncertainties related to the contaminant transport in unsaturated zone of Saligny site by:

- obtaining site specific data that accurately describe the contaminant interaction with geological media beneath the repository
- improving and validating the transport numerical models used in disposal safety assessment
  - develop new in-field tracer test and collect more experimental data for better understanding of the Saligny site characteristics
  - increase confidence in the numerical modelling used in radioactive waste disposal
- improving the integrated system for safety assessment of LIL waste disposal applied to Saligny site as an useful tool for further application in the repository licence.

To achieve the project objectives the experimental and modelling activities are performed in three phases.

The *phase I* (01.07 - 30.11.2014) is dedicated to development of the methodologies to be applied in the laboratory sorption tests, optimizing the experimental conditions for the in-field tracer test and development of the 3D grid for water flow and contaminant transport in Saligny vadose zone to be used in tracer test modelling. In order to collect new site hydrogeological data the monitoring network set up on the Saligny site will be inspected and appropriate measure to fix it will be taken.

Due to high quantity of concrete used in a surface disposal facility (structure of the disposal cells, waste immobilization) the contaminant plume potentially released from the disposal zone will have a high pH (12.5 -13). This could affect the sorption capacity of the geological media beneath the repository, especially in the first layers until the natural groundwater will dilute the plume released and decrease the pH. For this reason, laboratory experiments will be performed during the *phase II* (*01*.01 - 30.11.2015) of the project to assess the pH influence on the contaminant transport on radionuclide sorption. Also, during this phase an adequate tracers cocktail will be injected in Saligny unsaturated zone and tracer's concentration will be monitored in time to get enough data for a better understanding of the contaminant behaviour in the unsaturated zone and to bring more experimental data for validation of the 3D code for the transport models developed by CEA and integrated under the Alliances platform. Saligny specific meteorological and hydrogeological data will be collected using the monitoring network that has to be put in work when the tracer test will be launched. Based on site specific data, the codes for water flow will be validated.

In the last phase of the project (01.01 - 30.06.2016), the laboratory and in field experiments will be completed and the implication on Safety Case of the pH influence on radionuclide retention will be evaluated. The modelling activities will be focused on computer codes validation for contaminant transport, dose assessment for normal evolution scenario of Saligny LIL waste repository and sensitivity and uncertainty analysis in Alliances and GoldSim.

The main foreseen result of the project is an update of the integrated safety assessment for the Saligny LIL waste disposal. All components of the disposal system (multilayer cap, disposal area, unsaturated and saturated zone of Saligny site) will be modelled using Alliances Platform based on the actual disposal design and updated site characteristics. The integrated system for safety assessment will be flexible in order to accommodate future improvements in the site characterisation and will represent a useful tool in the safety assessments required for repository licensing.

This project is a continuation of the collaboration between ICN and CEA established under NSRWAD project (2010-2013) and opens real perspectives for further cooperation in the field of radioactive waste disposal. For instance, the licensing process of the LIL waste repository at Saligny offers clear opportunities for a future cooperation in the assistance of AN&DR to achieve its objectives.

Implementation of geological disposal is a key issue in the nuclear energy development and still offers large space for new collaborative research projects investigating and modelling uncertainty on the long term processes occurring in the complex system of radioactive waste final disposal.

At international level, there are also other countries concerned by the radioactive waste disposal problem and requesting assistance for safety assessment. The experience achieved under this project could be helpful in potential joint tenders.

The project achievements will be presented in international conferences and in common publications, but also on the project website: www.nuclear.ro/en/NSRAWD2.php

Project webpage address: www.nuclear.ro/en/NSRAWD2.php

Project C4-07

Increasing the lifetime of W coated CFC, graphite and CuCrZr tiles for long time exploitation as Plasma Facing Components for *WEST* project

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Within the framework of this project we proposed to extend the lifetime of W coated Plasma Facing Components (*PFC*) designed to *WEST* project. The *WEST* (*Tungsten Environment in Steady state Tokamak*) project is aimed to study and to supply technological solutions for *ITER* (*International Thermonuclear Experimental Reactor*). In this respect the *WEST* project intends to transform the *Tore Supra* tokamak into a platform for testing the *ITER* technologies especially by transforming the actual divertor into an actively cooling divertor similar to *ITER*. The materials used within the project will be similar to the ones that will be used at *ITER*.

Our goal is **to develop and test different methods to refurbish the W coatings deposited on substrates similar to those used in *WEST* reactor.** In this respect Combined Magnetron Sputtering and Ion Implantation (*CMSII)* technique will be used to coat with W, samples made of materials similar to those used in *Tore Supra* reactor: *N11 Carbon Fibre Composite (CFC)*, *Fine Grain Graphite* (*FGG*) and *CuCrZr* alloy. The coatings (mono-layer or multilayers) will be applied on both actively cooled and inertial components.

The project will have four parts:

I. The first part is dedicated to the deposition of W coatings on samples made of *N11 CFC*, *FGG* and *CuCrZr* alloy, materials used in the structure of the *WEST* reactor. The deposition will be performed by *INFLPR* (Romanian partner in the project). Selection of coatings layout will be done together with *IRFM* (French partner in the project).

At this stage a complete characterization of the coatings and an evaluation in terms of the resistance to high heat fluxes will be made (**Milestone 1**). The characterization of the coating before High Heat Flux Tests (*HHFT)* will give an "initial state" for each coating. That will be considered as a reference to achieve for the third part of the project.

II. After the *HHFT* and the identification of the failure mechanisms under thermal loading/stress and the definition of an (un)acceptable limit for each coating, the second part of the project will focus on the removal of damaged areas (**Milestone 2**). Different removal methods (milling, sand blasting etc.) will be tested.

III. The third part of the project will be focused on the re-deposition of new coatings on substrate materials recovered by method selected in second part of the project. The same three types of substrate materials (*N11 CFC*, *Graphite* and *CuCrZr* alloy) will be used for this new re-deposition.

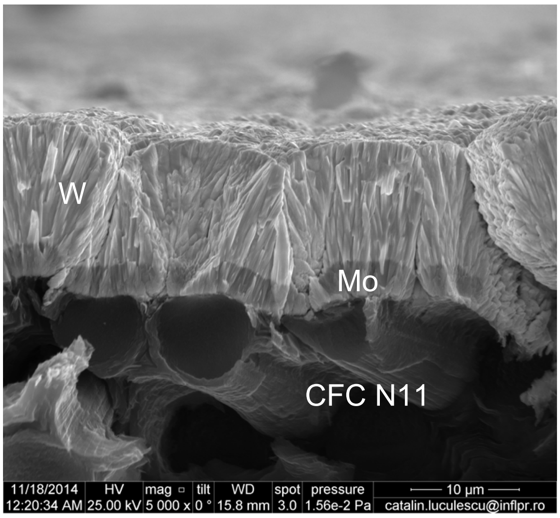
IV. The last part of this project will be focused on **the validation of the removal and deposition processes on real prototypes with complex geometry** (rounded edges and gaps). After removing the W coating, a complete analyze will be performed (on the surface and on the gaps) to assess the cleaning process and verify the absence of residues in the gaps. Then, a re-deposition of new coating will be performed and characterized.

In the first phase of the project the following objectives were addressed:

- Synthesis of W coating on *WEST* materials substrates
- Investigations of physical and chemical characteristics of deposited layers
- Adhesion assessment of W coatings deposited on *WEST* substrate materials

Results

W coatings with thickness of 14.5 μm have been obtained at this stage of the project progress. The coatings have been deposited on substrates similar to materials used in the *WEST* project namely: Carbon Fibre Composite type *N11*, Fine Grain Graphite (*FGG)* and *CuCrZr* alloy. Scanning Electron Microscopy (*SEM*) investigations revealed a columnar structure of the coatings (*Fig. 1*) and confirmed the coating thickness determined by Glow Discharge Optical Emission Spectrometry (*GDOES*) analysis.



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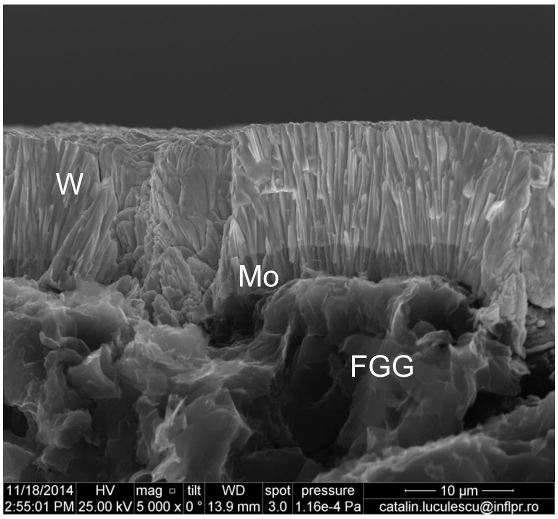

b

Fig.1 *SEM images of the W coatings deposited on* CFC N11 *substrate (a) and* FGG *(b) substrate respectively*

As far it concerns the influence of the coating process on the initial roughness of the substrate, no significant influences has been evidenced. Adhesion measurements performed on W coatings deposited on *CuCrZr* alloy indicated a critical load of 67 N. For a Ti witness samples coated with W in the same deposition run, the critical load exceeded 100N.

Some results of the project have been presented in the paper entitled "*The structure of the tungsten coatings deposited by Combined Magnetron Sputtering and Ion Implantation for nuclear fusion applications*", authors E. Grigore, C. Ruset, M. Rasinski, M. Gherendi, G. F. Matthews. This paper has been presented at SOFT 2014 Conference San Sebastian, Spain. The paper was submitted to Fusion Engineering and Design (Manuscript Number: FUSENGDES-D-14-00571).

The project page is hosted on the Plasma Surface Engineering Group web page.
Project webpage address: www.pse.inflpr.ro/cea-ro

Project C4-08

Climate change teleconnections between Western and Eastern Europe based on speleothem records of the Last Interglacial in France and Romania

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French project leader: **Dr. Dominique Blamart**
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Predicting the magnitude and effects of future global warming is crucial, among others, to mitigating the impacts on energy production and consumption. If one has to forecast climate developments in a warming world, one has to study an analogue to the Holocene (the current interglacial period) which presumably experienced warmer temperatures than those of today. Such an analogue is the Eemian, the Last Interglacial, which reached its warmth peak around 125 ka.

This project proposes to:

- detail climate evolutions at regional and European scale during the Last Interglacial using speleothem proxies from France and Romania;
- compare the Last Interglacial and Holocene climate dynamics at both regional and European scale.

For this, we will obtain high resolution, accurately dated time series of speleothem climate proxies from caves in Romania and France, covering the Last Interglacial. We are also continuously monitoring local environmental factors in Romanian and French caves in order to better understand transfer functions of climate information between the surface and the underground environments. Finally, we will perform time series analysis of the newly produced geochemical records in order to identify relevant periodicities in related to climate parameters. A preliminary result is shown in Fig. 1 and represents $\delta^{18}O$ and $\delta^{13}C$ time series of the CG1 stalagmite (SW Carpathians), compared to other regional datasets such as pollen-derived precipitation amounts from Velay (France).

The project involves frontier research and has an inter-disciplinary approach which gathers researchers from various fields of the Earth sciences: geology, geochemistry, geophysics, geomorphology, physics of the atmosphere, climatology. The results we foresee to stem from this project are linked to a better understanding of interglacial climate dynamics at regional and continental scale and to the increased understanding of how the climate system would act at European scale in the future, in case of sustained warming. This will further aid in drawing better adaptation policies to climate change at both national and European levels.

The two institutions, ISER and CEA, are both interested in collaborating on climate related topics, especially on studying past climate dynamics. As CEA, together with CNRS, are already involved in the study of past climate changes and ISER has acquired important expertise in studying climate archives from caves, this collaborative project fulfils a natural need for furthering the knowledge of the European palaeoclimate. Finally, the common activities of the two partner institutions, ISER and CEA, will contribute greatly to the prospect of them participating in joint European projects in the future.

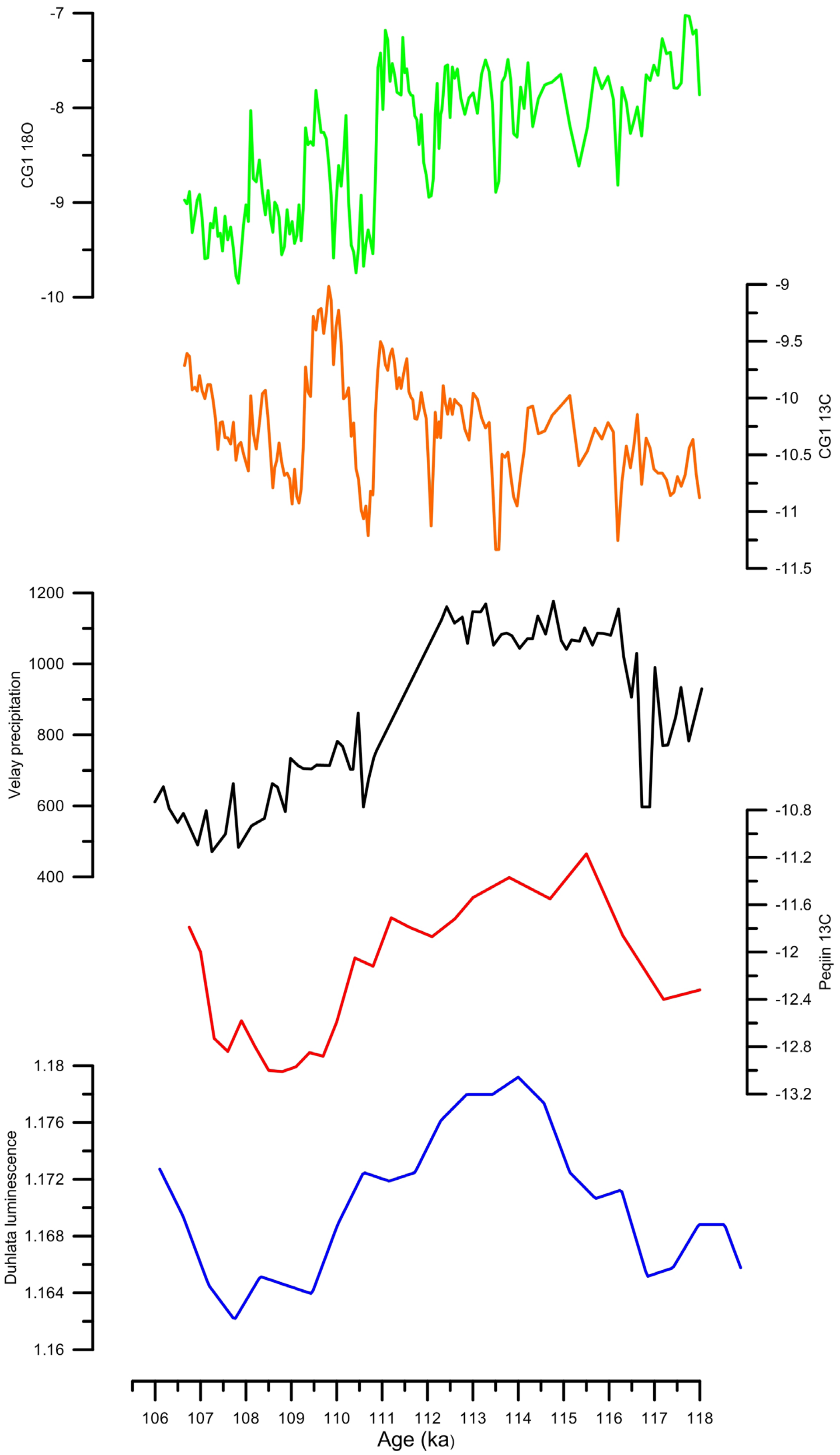

Fig. 1 – δ^{18}O and δ^{13}C time series of the CG1 stalagmite (SW Carpathians), compared to other regional datasets

Project webpage address: http://freem.iser.ro/.

Project C4-09

Energy for the future

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In the last years a lot of effort was concentrated in the studies related to the production of cleaner, safer and more reliable energy using new generation reactors. The present project aims to bring new and valuable knowledge using the NFS (Neutrons For Science) facility that is actually built at GANIL, France. The NFS facility will consist of a pulsed neutron beam-line and will be built in the first phase of the SPIRAL2 project and will be very competitive in terms of average flux relative to other facilities between 1 and 35 MeV. Moreover, NFS presents some distinct advantages due to the neutron production mechanism. In spallation neutron sources the high energy neutrons (up to hundreds MeV), may result in challenges for both collimation and background. The gamma-flash, which is known to be very detrimental to system dead-time that can effectively blind the detectors for the highest energy neutrons, is expected to be strongly reduced at NFS. Only very limited data exist for neutron induced reactions above 14 MeV. For many cases both fission and (n,xn) reaction cross-sections are unknown. The neutron energy range between 1 and 40 MeV concerns the nuclear waste transmutation in the case of ADS, future fusion applications, etc., where designers need new and good quality data and relevant codes in order to build evaluated data libraries and also to improve theoretical models.

The general objective of the project is to contribute to the development of new generation reactors by measuring cross sections for neutron induced reactions which are of interest for both Romanian and French teams in the field of fundamental and applied research in neutron physics. In the context of the Generation IV nuclear reactors studies, a lot of new reaction cross section measurements are in progress. The aim is to increase the level of the experimental nuclear data accuracy in view of improve the quality of simulation code predictions. In fast reactors, compared to conventional reactors (thermal), the (n,xn) reactions (with $x>1$) become possible despite their high threshold and play a non-negligible role, since above 10 MeV they have cross sections comparable to fission. They modify the neutron spectrum by converting fast neutrons ($E_n>5$ MeV) into slow neutrons ($E_n<1$ MeV) therefore influencing the criticality of the core. Furthermore, a rapid bibliography study shows that the (n,xn) reactions are not well known from the experimental and theoretical point of view. Three methods allow the measurement of these (n,xn) cross sections. The direct detection of the neutrons, the activation technique which is possible only if the final nucleus is unstable and has a life-time in a convenient range and the prompt γ spectroscopy. We have been involved in the development of an experimental set-up [1] based on this last method and measurements of (n,xn γ) cross sections for several nuclei have been already done [1,2] at the GELINA facility, Geel, Belgium, where the maximum of the neutron flux is around 1 MeV.

One weak point of this last method is that one measures transitions in the final nucleus, so that the direct production of this nucleus in its fundamental state is missed. The total (n,xn) cross section has to be determined by a model calculation. A straightforward way to check these calculations is to measure the cross section of one reaction simultaneously by the two last methods. The NFS facility gives us the opportunity to perform such experiments on the unique possible case 90Zr(n,3n)88Zr. Indeed, 90Zr is the lightest stable isotope of Zr, and contributes to more than 50% to natural Zr. The ground state band of the 88Zr nucleus formed by the reaction has the spin-parity sequence 0+, 2+, 4+ and the transitions between these states have energies of 1057 and 1082 keV. The ground state is unstable with a half life of 83 days and its decay produces a 393 keV transition in 88Y with 100% probability. We thus propose to perform the measurement of the 90Zr(n,3nγ)88Zr* cross sections by the prompt γ spectroscopy method and the measurement, by the activation technique, of the EC decay of 88Zr GS in the same experiment. As we use activation measurements the quasi mono-energetic neutron beam produced by the 7Li(p,n)7Be*, is recommended. The mono-energetic part and the continuous part of the neutron spectrum will be separated in the prompt measurement through the time of flight determination at 20 m. This will help to correct the activation measurement. This correction will even greatly be simplified by the fact that the two measurements will be performed at once, i.e. with the same set-up, the same target and the same beam. For the gamma detection we plan to use GAINS (Germanium Array for Inelastic Neutron Scattering) [3], developed at the GELINA facility (IRMM, Geel). It consists of an array of eight HPGe detectors placed at 110° and 150° with respect to the beam. As a conclusion the NFS facility allows us to perform experiments in view of providing new relevant (n,xn) reactions cross sections for example those included in the OECD/NEA High Priority Request List [4]

The project will be organized into three main tasks:

T1: New experiments at NFS and simulation start-up

This activity will be the first in the project and, from the justification of the proposed experiments to the choice of theoretical models to be implemented in the simulations. A very important aspect is related to the simulation of the background in the detection area. A status report of the project, at the end of first year, will present the results of this activity that will form the basis of the work to be performed in subsequent years.

T2. Simulation of physics cases with NFS

In this phase the majority of the simulation work will be performed using the conclusions reached during the T1 task. Simulations will use the Geant4 toolkit developed at CERN and the general purpose Monte-Carlo radiation transport MCNPX code will also be necessary to evaluate the neutron and gamma background for reactions at NFS. The results of the conceptual design and the physics simulations for GAINS array at NFS will be presented in the report at the end of the second year of the project.

T3. Experiments at NFS

By the end of 2015/beginning of 2016 we expect to run the first experiments at NFS and we expect that our experiment will be one of the first scheduled. This task consists in experiment preparation including GAINS detector installation, electronics mounting and settings, data taking, analysis and interpretation. These activities will be presented in the second year of the project as well in the final report.

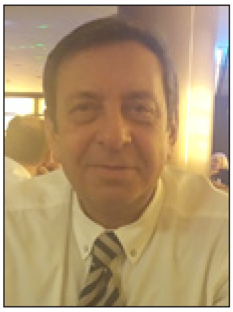
References:

1. *M.Kerveno* et al, Phys.Rev. C 87, 024609 (2013)
2. *J.C.Thiry* et al, J.Korean Phys.Soc. 59, 1880s (2011)
3. *D. Deleanu et al, NIM A 624 (130-136) (2010)*

Project webpage address: http://www.oecd-nea.org/dbdata/hprl/index.htm

Project C4-12

Markers implant for determining erosion and deposition effects on PFCs used in the first phase of the WEST PROJECT – CEA



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The objectives of the project are emerging form the WEST PROJECT (WP). Since one of the main objective of the WP is to study the ITER baseline H mode over long periods of time, by tackling in particular the problem of controlling the contamination of the plasma caused by tungsten, one main task is the characterization of the deuterium retention in Tungsten materials. This represents an important research task together with the characterization of erosion and deposition effects on the Plasma Facing Components (PFCs) installed in the French Tokamak Tore Supra.

In the frame of this project, erosion and deposition of different materials used in the Tore Supra will be measured quantitatively by use of energetic implanting of ions used as depth markers into specialized testing samples that can be afterwards mounted onto the PFCs. The remnant length of the marker or its depth positioning reveals the value of erosion or deposition on the sample material. The measurement of the deuterium retention is performed by use of a special method for concentration depth profiling, based Accelerator Mass Spectrometry (AMS).

The project activities were subdivided in two phases. First, investigation for finding optimum conditions for the implant of markers in PFCs used in the first phase of WEST. Second, phase is the determination of erosion/deposition by identification of the markers positioning and dimensions. Quantitative measurement of deuterium retention and inventory in tungsten PFCs of Tore Supra will be performed in 2017, when the first dismount of PFCs are foreseen.

In more detail, until October 2104, tests of different ion species for producing depth markers in tungsten was the first scientific task and deliverable. In this respect energetic implants of Si ions in W and glassy Carbon were tested. In a first experiment, implant energies for the accelerated ions were 1.335 MeV. This relatively low energy was chosen in order to obtain implant depths useful for the RBS analyses (hundred of nanometers). The obtained average implant depth was 480 nm for Si in W and 1100nm for Si in glassy carbon. The implanted ion dose was 4,7 10^{15} atoms/cm^2

The second task, subsequent to deliverable 1, was to perform until December 2014, Rutherford Backscattering (RBS) and Proton Induced X-ray emission (PIXE) measurements for the evaluation of results of the implanted W and glossy C samples. In figure 1 the RBS spectra are presented for the implant of Si ions in a glassy Carbon substrate.

For the ^{28}Si ions a peak can be distinguished in accordance to the simulation of the SIMNRA cod. The ^{16}O peak is present in an implant of ions. This is due to the migration of the oxygen from the sample surface where it is fixed by different oxides, into the bulk as free ions. Unfortunately, RBS experiments could not determine the implanted Si ions in the W substrate. This is obviously due to the heavy mass substrate (W) in comparison with the implanted/recoiling light ion of Si.

At this moment, experiments are in progress to establish the optimum method for detecting the implanted depth marker in tungsten material. The evaluated methods are AMS and SIMS. Investigations are extended also about the type of ion to be used as marker in tungsten and increase of the implanted ion dose, which at the moment was not exceeding 2 x 10^{16} atoms/cm^2.

The results will be used in the next tasks (deliverables 3and 4) consisting in the implant different markers in the provided samples, until September 2015 and the evaluation of the marker implant until October this year.

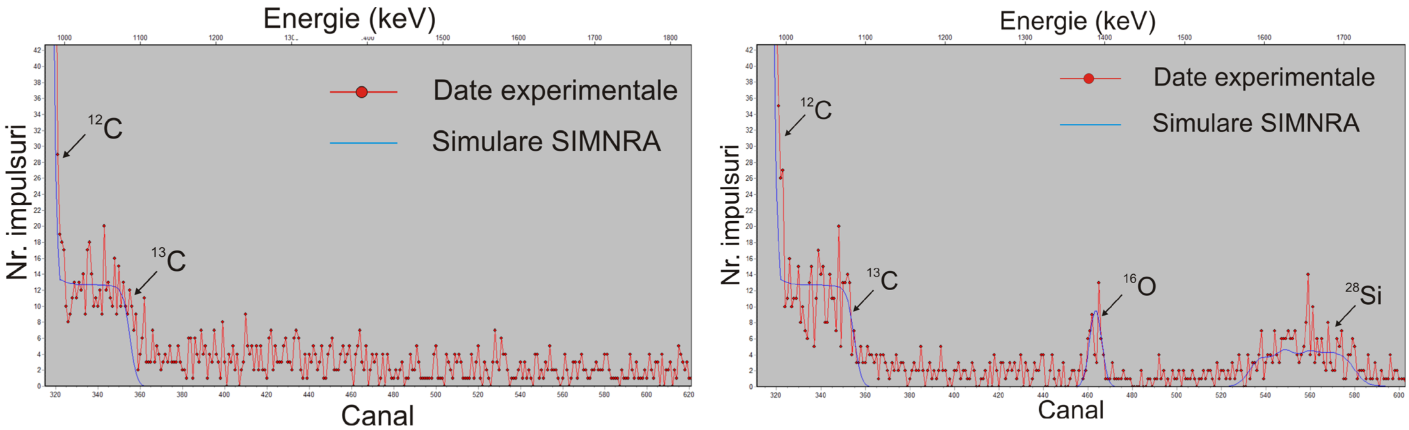
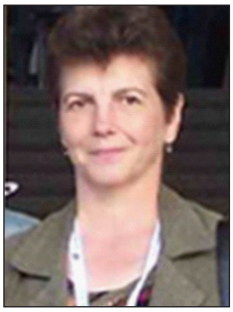


Fig.1 *RBS spectra form a carbon "glassy Carbon sample" implanted with 1.335MeV Si-ions. Left: before implant. Right: after implant with Si-ions. Simulations were carried out with the SIMNRA cod.*

Project C4-13

Dust mobilization and collection by laser and plasma



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Energy is the single most important commodity that has been a key in the development of human society. In the present day there is virtually no field of human activity, from agriculture to information technology, which is not reliant on the use of energy. The future operation of the ITER reactor at Cadarache is a great commitment and a monumental undertaking meant to address efficient and responsible production of energy. The scale and complexity of this project are hard to express in simple words, and yet all the aspects that comprise it are critical to its success.

The successful mobilization, transport and collection of dust created as by-product during the operation of the tokamak reactor is an important piece of the puzzle, and we think that the framework of the IFA-CEA programme presents an excellent opportunity to achieve a significant progress, beyond the state-of-the-art, in tackling this issue. As such, we propose a complex, interdisciplinary approach that combines elements of pulsed laser particle mobilization, plasmas and electric fields assistance in order to explore mechanisms for the mobilization, transport and collection of dust/powders from various types of surfaces.

We will check if the laser irradiation, plasma jet sources, and laser beams coupled with an incident plasma source are viable methods to achieve particles mobilization and transport. These experiments will also be carried out in electric field to see if ionized particles can be ultimately collected.

While our effort will revolve mainly around the treatment of fusion energy powders, it can also yield relevant results concerning the removal of dust from sensitive surfaces, such as those used in micro-devices for energy storage and production. Specifics related to the proposed materials, techniques and analyses are detailed in the subsequent sections of this proposal.

The project's work plan has three stages spanning over a period of 24 months. It comprises four work packages that will focus on: i) Production of particles and surfaces with particles by lasers and plasmas (WP1), ii) Mobilization, transport, collection of particles from contaminated surfaces (WP2), iii) Analysis of collection efficiency, material characterization (WP3), and iv) Dissemination of results through scientific papers and conference participations (WP4).

The greatest contribution that this project will bring is an in-depth understanding of the mechanisms governing dust mobilization, transport and collection employing a variety of laser and/or plasma techniques. This is expected to have a significant impact on the successful operation of the ITER tokamak, which is a next-generation fusion energy reactor requiring a non-invasive approach to the removal and handling of the large quantities of tritiated dust expected to be produced throughout its operation. If no successful process is found, the operation of ITER could be constraint due to in vessel activities needed to recover these small particles by sucking under atmosphere.

Economically, the financial effort dedicated to funding such a project is very small when compared to the potential benefits stemming from finding a plausible approach to solve one of ITER's critical operability issues. The collective results of the project would impact not only the operability of the ITER facility, but they will also contribute to an environmentally friendly solution to the subsequent handling of these hazardous by-products. Changes brought about to their chemistry through nitrating or reducing of the powders by laser and plasma techniques also impact the issue of their subsequent disposal. It should be noted that laser and plasma techniques are clean themselves, and do not pose additional stress on the facility, from an environmental perspective.

As the first stage of the project has ended, we can now draw some relevant conclusions regarding the realization of surfaces contaminated with metal powders by using laser and plasma techniques. For instance, W particles having sizes between 70-100 nm have been synthesized using a cluster source that combines magnetron sputtering and condensation of metal vapours in inert gas - MS-GAS. This proved to be a very efficient approach towards the production of high quantities of metal powders (see inset in Fig. 1) having a narrow size distribution. On the other hand, a plasma jet method, suitable for making particles at atmospheric pressure was developed. A relevant image is presented in Fig.1. In addition, laser-based methods (pulsed laser deposition - PLD, ablation of a metal target in liquid medium) have yielded surfaces having a much lower particle density, which would be better suited for fundamental studies (Fig. 2).

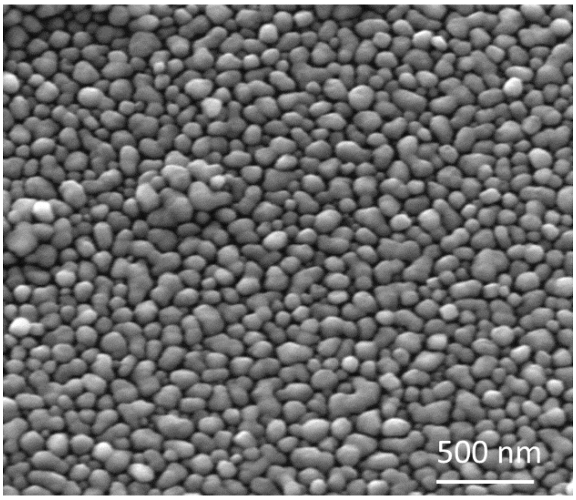

Fig. 1 SEM image of particles produced by plasma jet surface obtained by PLD

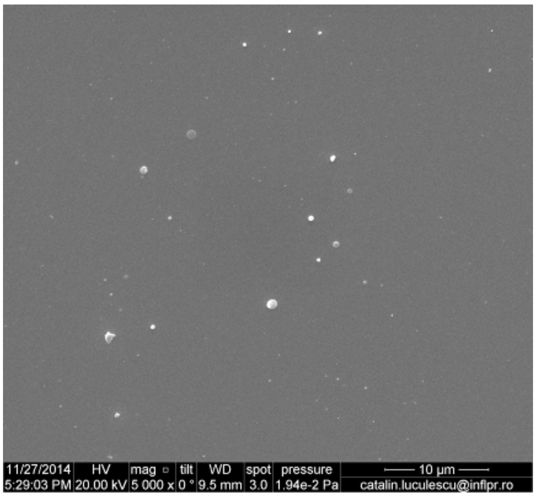

Fig. 2 W contaminated

The next step of our research agenda is to use the obtained samples to investigate the influence of the type of bonding between particles and surface on the removal efficiency of the particles.

We foresee that a successful development of the objectives will strengthen the cooperation between the institutions involved in the project by opening up perspectives for future collaborations with an industrial partner having engineering capabilities in the framework of EFDA and Horizon 2020 research programmes.

Project webpage address: http://ppam.inflpr.ro/DUSTCO.htm

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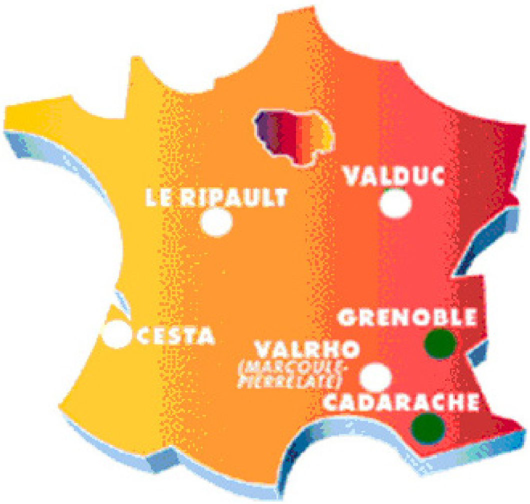

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