Title: Improving capabilities for the applications of positron annihilation spectroscopy

Original Language Title: Poboljšanje mogucnosti za primjenu pozitronsko anihilacijske spektroskopije

Project Number: CRO/0/009

Project Type: National

Project Class: Category A

Submitted By: Member State and/or Observers With Rights

Field of Activity: 01 - Building nuclear science competencies

FOA Distribution:
FoA Code: 01 = 100%

Link to RB Programme: There is No RB Programme Link.

Participating Member State(s):
Croatia

Project duration (Total number of years): 2

Project duration (Start date): 2014-01-01

Overall Objective: To increase utilization of nuclear analytical techniques in industrial applications for products of benefit for overall population by improvement of positron annihilation spectroscopy analytical services in the laboratory at the Department of Physics, Faculty of Science, Zagreb

Objective Analysis: Upgraded laboratory will provide analytical services using positron annihilation spectroscopy (PAS) to the industrial partners in the country and region for the control and inspection of their products and production processes. Specific PALS and DBS systems with appropriate time resolution, time range, energy resolution and sample storages needed for different types of samples will be established and personal will be trained. These analytical services will be possible to use in the control and inspection of broad spectrum of materials of high technological importance (e.g. membranes for water purification and desalination, zeolites and mesoporous materials, solar cells, cement-based materials, various polymers, various metallic alloys, various ceramics, radiation or mechanical stress damages in materials, etc.). The introduction of these analytical service will also enhance academia-industry cooperation and transfer of knowledge, and that is one of the strategic goals of University of Zagreb and the country in general (please see support letter from the University of Zagreb, Support-University-of-Zagreb.pdf). Eventually this will improve production processes and products quality, enhance competitivity and lead to production of better products for the benefit of overall population (e.g. water supply, energy production, electronics, environment protection, etc.). Successful employment of these analytical services addresses also one of the listed priority topics of TC programme in Country Programme Framework - “development and applications of nuclear techniques”. Developed objective tree is schematically presented in the attached diagram – ObjectiveTree.pdf.

Gap / Problem / Need Analysis: Nuclear methods have already proved themselves as very valuable analytical tools in various fields, and among them positron annihilation spectroscopy (PAS), as a non-destructive method is playing prominent role targeting nano-scale region in control and inspection of broad spectrum of different, technological high important materials and products of benefit for overall population. Positron annihilation spectroscopy techniques (specific techniques in this project include positron annihilation lifetime spectroscopy, PALS, and Doppler broadening spectroscopy, DBS) enable us to acquire high precision information on electron structure, void and defect patterns in an extremely broad spectrum of ordered and disordered materials, in particular information on the disordered, amorphous structure of polymers, pores in catalysts, atomic vacancies in metals and semiconductors, that are inaccessible by other techniques, and make them very valuable for inspection and control of specific products and production processes. In Croatia there is no laboratory that can provide such analytical services. Initially started applications of positron annihilation lifetime spectroscopy by using system built at the Department of Physics have shown potential of the method and have attracted the interest of potential industrial counterpart for the control of their products, but very restricted capabilities (analytical properties and also time availability) of the system does not allow employment of this method in its full power. The lack of these analytical services prevents their efficient and successful employment in industrial applications of control, inspection and optimization of broad spectrum of products of general interest. This also prevents stronger collaboration between academia and industrial partners, one of the strategic goals of the country that is presently either weak or not existing, especially in the field of industrial applications of nuclear techniques.

Role of nuclear technology: Two techniques of positron annihilation spectroscopy will be used: positron annihilation lifetime spectroscopy and Doppler broadening spectroscopy, with 22Na as a positron source. Positron annihilation lifetime spectroscopy is the unique nuclear non-destructive technique that gives information about nano-scale void distributions in the investigated sample by measuring the lifetime of positrons in the sample as a time difference between appearance of initial and annihilation gamma rays. After emission of positron from 22Na source, 22Ne is produced in an excited state and it emits almost instantaneously one gamma ray of 1.27 MeV, start signal. Emitted positron enters the sample, thermalizes, and annihilates with an electron with the emission of two gamma rays of approx. 0.51 MeV, stop signal. If the voids, of different origin, are present in the sample, they can extend lifetimes of positrons, and that gives information about the sizes and relative distributions of voids. The sizes of the voids can be from several to several tens of nano-meters. There is no other nuclear or non-nuclear technique that can give similar information. This is also valid for Doppler broadening spectroscopy, that can also give information about chemical content at the site of annihilation by measuring the energy variations of annihilated gamma rays. The limitation of the analytical techniques with 22Na source is on the possible size and thickness of the investigated sample and this is determined by the energies of the positrons emitted from the 22Na source.

Physical infrastructure and human resources: The experimental nuclear physics group at the Department of Physics has a laboratory space of 75 m2 at its disposal (plus lab space of 50 m2 devoted for teaching), and tree offices of 25 m2, each. At the Department there already exists nuclear physics equipment, but that is mainly used for teaching purposes. Small part of the time (approx. 3-4 months per year) this equipment is available for other purposes, and in that time preliminary investigations and studies of the potential of positron annihilation spectroscopy systems and preliminary investigations of specific samples have been performed (using PALS system based on CAMAC electronics and one-arm DBS system). The experimental nuclear physics group consists of one professor (Damir Bosnar), one docent (Mihael Makek), one research associate (Gorjana Jerbic Zorc) and two graduate students (Ivica Frišcic and Petar Žugec), who will participate in the project. Damir Bosnar has more than 25 years experience in the experimental nuclear physics, he has been working in basic research in experimental physics participating at the experiments at Paul Scherrer Institute, Switzerland, Mainz Microtron, Germany, Laboratori Nazionali di Frascati, Italy and CERN, and more than 70 publications have resulted from these investigations. At the Department of Physics he has been the leading person in the upgrade of laboratory for nuclear science and applications. In the laboratory he has built PALS system based on conventional analog electronics with the timing characteristics comparable to the commercial systems, but with additional feature of gama-ray energies recording (D. Bosnar et al Nucl. Instr. Meth. A581 (2007) 91). The employment of the system in the analysis of various samples (zeolites, membranes, foils, metals, alloys) has been started. In cooperation with G. Jerbic Zorc, one arm Doppler broadening system has been built and initial analysis of some zeolites and metals have been started. The members of the group have large experience with fast pulse digitizers and digital signal processing, that they employed in various nuclear physics measurements (M. Makek et al, Nucl. Instr. Meth. A673 (2012) 82,

P. Žugec , Nucl. Inst. Meth. A659 (2011)307, D. Bosnar IAEA-TECDOC-1634, Proceedings of Technical Meeting, Vienna 2007, p. 29). The members of the group have also large experience in building and tuning both simple and more complex detector and electronics systems for nuclear physics measurements and applications. For example, at the Department they have built PET model consisting of 48 BaF2 detectors. Detectors have been assembled from the purchased commercial components (BaF2 scintillators, PMTs and VDs) and from the supporting mechanical parts machined at the Department, with data processing and acquisition based on CAMAC and VME electronics system (I. Frišcic. M. Makek, D. Bosnar, M. Distler, Coincidence and time determination in ToF PET model with TDC in continuous mode, 12th Vienna Conference on Instrumentation, Vienna, 2010). Beside the research and applications of nuclear methods, the members of the group have introduced various student lab exercises in nuclear physics and applications at the Department, and several courses with appropriate exercises have been introduced both in the undergraduate and graduate studies. These education activities will also secure qualified staff for the applications of nuclear techniques in the future.

Safety regulatory infrastructure: There exists all necessary safety regulations and existing nuclear physics laboratory already obeys all regulations.

Stakeholder analysis and partnerships: Target groups are service users in high-tech oriented SMEs and industry, present and future, in the country and in the region, which can benefit from specific analytical services in the laboratory. One of the strategic goals of the country is to establish and increase collaboration between academia and industry. Our project will contribute to that by introducing and implementing nuclear techniques in industrial applications, especially in present and future SME. In this sense the proposed project is strongly supported by the University of Zagreb as fulfilling one of the strategic goals of the University and the country in general (support letter Support-University-of-Zagreb.pdf) . Some of the examples of possible applications of the positron annihilation spectroscopy analytical and inspection services for industrial partners include: zeolites and mesoporous materials, membranes for water purification and desalination, solar cells, cement-based materials, various polymers, various metallic alloys, various ceramics, inspection of radiation or mechanical stress damages. List of potential stakeholders interested in control of specific products and production processes includes companies producing: water purification and desalination membranes, foils for liquid (food) packages, solar cells, semiconductors, metals and alloys, etc. The Ministry of Science, Education and Sports, that can be considered as a stakeholder representing overall population as final beneficiary of the project, is also strongly supporting the proposed project (please see the support letter Support-Ministry-of-Science.pdf). The proposed, application oriented, project is complementary project to the currently existing scientific research project financed by Ministry and led by the project proposer ("QCD and nuclei; applications of nuclear methods: materials, medicine and environment"). The list of identified individua stakeholders, their interest and possible impact is given in the attached table - Stakeholder.pdf. As for partnership, in the preparation phase of the project there was cooperation, mainly research oriented, with several national institutions in the sample preparations for PALS and DBS measurements (V. Dananic from Faculty of Chemical Engineering and Technology in Zagreb, S. Valic from Department of Chemistry and Biochemistry, University of Rijeka, S. Bosnar from Rudjer Boškovic Institute Zagreb,). With them strong cooperation has been established and also initial links with industrial counterparts in the inspection and control of their products have been created. These partners will continue to collaborate and participate particularly in the sample preparations needed for system calibrations. At the international level there has been cooperation with KFKI and Mediso Ltd, Budapest, in the optimization of the PALS system and design of mechanical support for the detectors and samples. It is expected that they will continue to collaborate in the project during the setup of the positron annihilation systems (expert visits, scientific visits, fellowships). There was also collaboration with the Institute for Nuclear Physics in Mainz in the part of optimization and tuning of the electronics for the proposed systems and it is expected that they will collaborate in this part in the proposed project (expert visits, scientific visits, fellowships).

Other considerations, e.g. environment, gender: In the execution of the project there is no direct influence on the environment and there is no possibility of any negative influence on the environment. But, the outcome of the project should be analytical systems for the inspection and production control of the products that can have positive impact on the environment, e.g. water purification and desalination, energy production (various membranes, zeolite systems, etc.). The results of the project will be of equal benefit for both men and women. Female researchers are included in the project both at the Department of Physics (one research associate) and in the partner institutions (one research associate).

Implementation strategy: In the first year two PALS systems with different characteristics will be established: one with very good timing resolution and one with wide time window. In the second year coincidence Doppler broadening system will be established. The project coordinator will coordinate these activities. Although there exist commercially available systems (e.g. Ortec PLS-system for PALS, similar for DBS), our general approach will be to assemble our own systems from commercially available components: detector parts and electronic units and to machine necessary mechanical parts and supports at the Department. These are relatively simple systems to assemble and tune for qualified persons and we possess all required expertise and manpower for that (please see "Physical infrastructure and human resources"). And the most important reasons are prices and desired system characteristics. It can be easily shown that identical systems if assembled from commercially available components and tuned by ourselves are at least two times cheaper than commercially available whole system, with eventually the same characteristics or even better characteristics can be achieved. PALS systems will be based on fast pulse digitizers: all necessary components are well established and commercially available and system characteristics are eventually superior to the system based on analog electronics that is commercially available. Coincidence DBS system will be based on analog electronics, that is well established and proved spectroscopy system. Specific reference samples needed for the system calibration will be provided by the project partners. Additional personal training needed for the work with the system will be done in the framework of expert visits, fellowships and scientific visits.

Monitoring and progress reporting: - Technical characteristics of the established positron annihilation systems will be documented. - Analytical potential of the systems for different types of the samples will be documented. - The user manuals for the developed analysis software will be provided.

Risk management: There exists required lab space infrastructure for the realization of the proposed project at the Department of Physics, Faculty of Science, Zagreb. The project participants have required expertise and capacity to assemble and tune the proposed systems. Some specific items (e..g. in systems optimization, samples analysis) will be realized through expert visits, scientific visits and fellowships.

CORE FINANCING										
Year	**Human Resource Components** (Euros)						**Procurement Components** (Euros)			**Total** (Euros)
	Experts	Meetings/ Workshop	Fellow-ships	Scientific Visits	Training Courses	**Sub-Total**	Equipment	Sub-Contracts	**Sub-Total**	
2014	20 000	0	5 400	6 000	0	**31 400**	80 000	0	**80 000**	**111 400**
2015	15 000	0	5 400	3 000	0	**23 400**	68 000	0	**68 000**	**91 400**
First Year Approved : 2014										

Logical Framework Matrix (LFM)

	Design Element	**Indicator**	**Means of Verification**	**Assumptions**
Outcome	Improved positron annihilation spectroscopy analytical services	Positron annihilation analytical systems with	System description with technical characteristics	Acceptance of the analytical services

	for industrial counterparts and enhanced academia-industry cooperation.	desired properties that can provide services to broad spectrum of industrial partners producing goods of interest for overall population and enhancement of academia-industry cooperation.	of the established analytical system and list of available services on the web site of the laboratory and in the hard copy.	by the industrial end-users in the country and region.
Output	1 Project Management Team Operational			
	2 One PALS system with good time resolution and one PALS system with wide time window established.	Operating PALS system with good time resolution and operating PALS system with wide time window, by the end of the 1st year, and relevant control and inspection measurements for industrial partners possible.	Results of measurements of suitable reference samples obtained using PALS systems.	Required PALS system characteristics for timing and time window achieved.
	3 Coincidence Doppler broadening system established.	Operating coincidence Doppler broadening spectroscopy system, by the end of 2nd year, and relevant control and inspection measurements for industrial partners possible.	Results of measurements of suitable reference samples using the coincidence DBS system.	Required characteristics of the coincidence Doppler broadening system achieved.
	4 Trained personal for particular PAS system.	Trained staff for PALS analytical services by the end of the 1st year, trained staff for coincidence DBS analytical services by the end of 2nd year.	Analyzed and interpreted results of measurements of reference samples.	Appropriate training acquired.
Activity	1.1 Confirming/Setting-up project team (CP, CP team in MS, PMO/TO)			
	1.2 Conducting project review meetings			
	1.3 Updating project work plan			

	1.4 Preparing and submitting PPARs (every six months)			
	1.5 IAEA Field Monitoring			
	2.1 Assembling of PALS system with good time resolution.	PALS system with goof time resolution assembled by the middle of 1st year.	System description, user manual.	Required equipment acquired.
	2.2 Optimization of the PALS system with good time resolution and determination of its characteristics.	Optimized PALS system with good time resolution by the end of the third quarter of 1st year.	Analyzed calibration data.	System assembled, appropriate calibration source supplied, expert visit realized.
	2.3 Assembling of the PALS system with wide time window.	Assembled PALS system with wide time window by the middle of 1st year.	System description. User manual.	Required equipment acquired.
	2.4 Optimization of the PALS system with wide time window and determination of its characteristics.	Optimized PALS system with wide time window by the end of the third quarter of the 1st year.	Analyzed calibration data.	System assembled, appropriate calibration source supplied, visits realized.
	3.1 Assembling of coincidence Doppler broadening system.	Assembled coincidence Doppler broadening system by the middle of second year.	System description. User manual.	Required equipment acquired.
	3.2 Optimization and characterization of the coincidence Doppler broadening system.	Optimized coincidence Doppler broadening system by the end of third quarter of the second year.	Analyzed calibration measurements.	System assembled, calibration source acquired, visits realized.
	4.1 Training for the usage of the PALS with good time resolution.	Personal trained by the end of the third quarter of the 1st year.	Analyzed and interpreted results of measurements of reference samples.	Appropriate counterparts identified and available.

	4.2 Training for the usage of the PALS with wide time window.	Personal trained by the end of the third quarter of the 1st year.	Analyzed and interpreted results of measurement of reference samples.	Appropriate counterparts identified and available.
	4.3 Training for the usage of coincidence Doppler broadening spectroscopy system.	Personal trained by the end of the 2nd year.	Analyzed and interpreted results of measurements of reference samples.	Appropriate counterparts identified and available.
Input	2.1.1 Detector components, electronics for the PALS system with good time resolution: scintillation detectors, PMTs, HVs, very fast pulse digitizer (PCI type) with data acquisition software.	Equipment purchased in the first half of the 1st year.	Delivered equipment.	Appropriate equipment identified and ordered.
	2.1.2 Mechanical parts and supports. Cables and connectors. Computers. Osciloscope.	Equipment purchased and supplied by the first half of 1st year.	Delivered equipment.	Appropriate equipment supplied.
	2.2.1 Expert visit connected with the optimization of the PALS system with good time resolution.	Expert visit in the second quarter of the 1st year.	End of mission report.	Appropriate expert identified and accepts visit.
	2.2.2 22Na source, samples for calibration measurements.	Appropriate 22Na source, appropriate sample supplied.	Source and sample characteristics.	22Na source and samples provided.
	2.3.1 Detector components and electronics: scintillation detectors, PMTs, HVs, fast pulse digitizer with data acquisition software, computer.	Equipment purchased in the first half of the 1st year.	Equipment delivered.	Appropriate detector components identified and ordered.
	2.3.2 Mechanical parts and supports. Cables and connectors.	Equipment purchased and supplied in the first half of 1st year.	Equipment delivered.	Appropriate mechanical parts, supports, designed, cables and computer ordered.

	2.4.1 22Na source. Samples for calibration measurement.	Appropriate 22Na source and samples supplied by the end of third quarter of the 1st year.	Source and samples delivered.	Relevant 22Na source and samples identified.
	2.4.2 Expert visit connected with optimization of the PALS system with wide time window and calibration measurements.	Expert visit in the third quarter of the 1st year.	End of mission report.	Expert identified and accepts visit.
	3.1.1 One HPGe detector for the coincidence Doppler broadening system, HV, amplifier. Electronics components for the coincidence Doppler broadening system: NIM crate, discriminators, coincidence, ADCs, data acquisition software.	Equipment purchased in the 1st half of the 2nd year.	Equipment delivered.	Appropriate equipment identified and ordered.
	3.1.2 Mecahnical parts and support. Cables and connectors. Computer.	Appropriate components and equipment supplied in the first half of 2nd year.	Equipment delivered.	Mechanical parts and supports designed. Equipment ordered.
	3.1.3 One HPGe detector, HV, amplifier, computer.	Equipment supplied in the 1st half of 2nd year.	Equipment delivered.	Appropriate equipment identified and ordered.
	3.2.1 22Na source, samples for the calibration measurements	Appropriate 22Na source and samples supplied by the end of third quarter of the 1st year.	22Na source and samples available.	Relevant 22Na source and samples identified and ordered.
	3.2.2 Expert visit connected with the calibration measurements with the coincidence Doppler broadening spectroscopy.	Expert visit realized in the 2nd quarter of the 2nd year.	End of mission report.	Expert identified and accepts visit.
	4.1.1 Scientific visit connected with software implementation and data interpretation in the measurements with the PALS	SV realized in the 1st half of 1st year.	End of mission report.	Counterpart identified and available.

	system with good time resolution.			
	4.1.2 Fellowship in the training for data acquisition for the PALS system based on the fast digitizers.	Fellowship realized in the first half of the 1st year.	End of mission report.	Counterpart identified and available.
	4.1.3 Expert visit connected with the interpretation of measurements with the PALS system with good time resolution.	Expert visit realized in the third quarter of the first year.	End of mission report.	Expert identified and accepts visit.
	4.2.1 Expert visit connected with the interpretation of the measurements with the PALS system with wide time window.	Expert visit realized in the forth quarter of 1st year.	End of mission report.	Expert identified and accepts visit.
	4.2.2 Scientific visit connected with the background control and determination in measurements with the PALS system with wide time window.	SV realized in the 2nd quarter of the 1st year.	End of mission report.	Counterpart identified and available.
	4.3.1 Fellowship connected with measurements with PALS system.	Fellowship realized in the 1st half of 2nd year.	End of mission report.	Counterpart identified and available.
	4.3.2 Scientific visit connected with the specific data interpretation in measurements with coincidence Doppler broadening spectroscopy system.	SV visit realized in the 2nd quarter of 2nd year.	End of mission report.	Counterpart identified and available.
	4.3.3 Expert visit connected with the interpretation of the results of measurements with coincidence Doppler broadening system.	Expert visit realized in the third quarter of 2nd year.	End of mission report	Expert identified and accepts visit.