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HISTORICAL REVIEW OF NEUTRON SPECTROMETRY AND DIFFRACTION IN BRAZIL – 50 YEARS

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ABSTRACT

Neutron Spectrometry and Diffraction techniques began to be studied in Brazil with the construction of the first research reactors in the years 50 and 60, and were developed with the participation of national and international scientists. In particular, we note the perseverance of the Brazilian pioneer of nuclear energy, Dr. Marcello Damy de Souza Santos✝, and from that time, with the involvement of some of the early pioneers, such as the physicist Roberto Stasiulevicius✝, who had the privilege of working in all three Brazilian nuclear research reactors. All these scientists shared half a century of acquired knowledge, helping in the training of a number of experts who are now working in national and international nuclear institutes. The present work reviews in chronological order, the testimony of the historical facts associated with the development and application of experimental techniques using neutrons as a tool for materials analysis and studies for many areas of the sciences and also with the training of personnel. IPEN, IEN and CDTN, all institutes of the CNEN-Brazil, are important in the national and international scene as a result of the research carried out there and the large number of publications produced in the field of neutron spectrometry and diffraction from experimental work carried out using the IEA (5MW), Argonauta (5kW) and more recently, the IPR (250kW) research reactors. An extensive bibliography is also presented and this can be disseminated to everyone interested.
✝Posthumous

1. INTRODUCTION

Nuclear techniques associated a radiation source as a nuclear reactor have importance for applications in characterization, analysis and control of quality of materials. Neutron spectrometry allows the determination of reactor neutron spectra, frequency spectra, scattering laws and neutron cross-sections of materials, all of which are important for nuclear and particle physics, engineering and reactor physics in the design of the nuclear reactor core and applications in other scientific areas as medicine, biology, radiopharmacy and dosimetry.

Neutron spectrometry allows information to be obtained about atomic and molecular structures ranging from those consisting of single atomic species to complex biopolymers. Neutron used as a probe for small samples of materials reveal what is difficult to study using chemical-physics techniques as NMR (Nuclear Magnetic Resonance), IR(Infra red), X-ray and others. Some example include isotopic information about atomic nuclei of materials, the control of nuclear purity of these materials, the control of H/D rates in molecules, H and C in hydrogenous and carbonated compounds, and others applications as in the study of the dynamics and structure of molecules and amorphous materials,.

Neutron diffraction can be used to determine the static structure factor of gases, liquids, or amorphous solids and is an important tool of crystallography aim at the structure of crystalline solids and in condensed matter physics with applications many others materials. Neutron has a magnetic dipole moment that makes it sensitive to magnetic fields from unpaired electrons in materials. It is a powerful tool for studies of magnetic and paramagnetic interactions in materials using diffraction techniques.

The single crystal version of the technique is less commonly used because currently available neutron sources require relatively large samples and large single crystals are hard or impossible to come by for most materials. Because the data is typically a 1D powder diffractogram, they are usually processed using the Rietveld refinement.

Practical applications of neutron diffraction include the determination of the lattice constant of metals and other crystalline materials, for which, with an accurately aligned micro positional a map of the lattice constant through the metal can be derived. This can easily be converted to the stress field experienced by the material. Neutrons are very sensitive to the presence of H in the molecular structure. This means that the position of hydrogen in a crystal structure and its thermal motion can be determined far more precisely with neutrons. Neutron scattering lengths of opposite sign for H and D allow a contrast variation high enough to highlight one element in an otherwise complicated structure. It plays an exceptionally large role in biochemical molecules and compounds that are difficult to study structurally in other ways.

This work provides a review in chronological order as a testimony to the historical facts associated with the development and application of the experimental techniques of neutron diffraction and spectrometry as a tool for materials analysis and studies for many areas of the sciences and also for the training of personnel. The experimental arrangements using the IEA (5MW), Argonauta (5kW) and more recently, the IPR (250kW) research reactors are presented. The IPEN, IEN and CDTN, all institutes of the CNEN-Brazil, are important in the national and international scene as a result of the research carried out there and the large number of publications produced in the field of neutron spectrometry and diffraction

2. HISTORICAL REVIEW

Neutron Spectrometry and Diffraction techniques began to be studied in Brazil with the construction of the first research reactor in the year 1956, the IEA-R1(5MW) in the old Instituto de Energia Atomica (IEA), now the Instituto de Pesquisas Energéticas e

Nucleares(IPEN-CNEN-SP) and with the construction of the Argonauta reactor(5kW) in the year 1965 at the Instituto de Engenharia Nuclear (IEN).

These techniques were developed with the participation of renowned national and international scientists, in particular, with the perseverance of the Brazilian pioneer of nuclear energy, Dr. Marcello Damy de Souza Santos✝ in the old IEA and from that time, with the involvement of some of the early pioneers, such as the physicist Roberto Stasiulevicius also in the IEA and the engineer Luiz Ozório de Brito Aghina and others collaborators in IEN.

In the IEA-R1 the first group of scientists was formed in the nuclear physics division managed by Dr. Damy that constructed the first national crystal neutron spectrometer, the EC-IEA, installed in a radial beam hole of the reactor, where the first experimental nuclear physics work using neutrons as a tool for nuclear measurements[1] took place. Total neutron cross-sections of water and many hydrogenous compounds such as polyethylene and metallic compounds were measured. The EC-IEA was also fundamental in the quality control of the uranium produced in the chemical division of the old IEA, nowadays as part of the ore reserves of the country.

In 1961 and 1962, the mathematician Marieta Camargo Mattos and the IAEA expert Dr.Robert Lee Zimmerman joined this group, followed in 1963 by Lia Queiroz do Amaral and Fernando Giovani Bianchini, physics students of Universidade de São Paulo(USP) and later researchers of the IEA.

For the determination of the fission neutron spectrum of the IEA-R1 reactor, an other spectrometer was constructed, a simplified proton recoil telescope by the physicist Silvio Bruni Herdade with the researchers S. Paiano, A. A. Suarez and Olga Y. Mafra[2] as collaborators. In 1964, the former was named manager of the nuclear physics division and he took a fundamental step in the design and construction of a neutron mechanical velocity selector (MVS) was constructed as a filter for high-order reflected neutrons from the monochromator crystal [3].

Neutron cross-sections of Ag, Al, C, Cu, Cd and many oxide compounds of rare earths such as Er, Ho, Lu, Yb, Pr and Tm were measured with the MVS incorporated in the EC-IEA. This data was published with merit and international distinction in the “Barn Book” BNL-325 of the BNL(Brookhaven National Laboratory) and also reported in IAEA publications [4].

In the same period with the collaboration of the researchers Lia Queiroz do Amaral, Claudio Rodrigues and Laércio Antonio Vinhas, a chopper with bent plates and time-of-flight/Be filter techniques was constructed.[5] and also a multi purpose neutron diffractometer was designed and constructed by the physicist and researcher Dr.Carlos Benedicto Ramos Parente with the collaboration of the USP Physics students K.Harada and Y.Koishi [6,7].

In 1972, the nuclear physics division began to be managed by the researcher Roberto Fulfaro who, with the collaboration of the the researcher José Mestnik Filho and others, constructed a triple axis spectrometer (TAS) for neutron scattering measurements [8]. The neutron diffractometer had continued to be improved by Dr.Parente and collaborators, mainly when joined to the group the physicist Vera L. Mazzocchi developing its doctoral thesis in 1994 [20]. In 1997 the physicist Roberto Stasiulevicius now working in IPR-CNEN-MG concluded his doctoral thesis about natural crystals applied to neutron diffractometry and espectrometry[21].With the large quantity of publications and the demand on it, the design of a new neutron diffractometer became necessary. In 2005, the neutron diffractometer PSD was constructed, opening applications in other fields of the sciences. It continues to be operated up to the present by Dr.Parente and collaborators. The chopper with time-of-flight/Be filter spectrometer has been de-activated but the TAS continues to be operated by Dr. José Mestnik Filho.

Neutron spectrometry in IEN was initiated in 1968 under the gestation of the old Companhia Brasileira de Tecnologia Nuclear (CBTN). The reactor division was managed by the engineer Luiz Ozório de Brito Aghina who was involved with the comparison and validation of the Sophocate code and the Nelkin model, both used in calculation of the reactor core.

For applications in the Argonaut reactor to thermal neutron spectrum determination, the researcher Luiz Pinguelli Rosa designed and constructed the first neutron spectrometer, a chopper with time-of-flight similar to that of the ANL(Argonne National Laboratory), which was also used some years later for the reactor thermal neutron spectrum determination by Getulio Jesus Vilar in his post-graduate thesis under the orientation of Dr John Douglas Rogers from COPPE-UFRJ.

Later, the researchers Frida Eidelman and Miriam Dias Pacheco developed the use of NE-213 recoil proton detectors for fast neutron spectra determination with the collaboration of the researchers Wilma dos Santos Bastos and Juan B. Soto Hesles, under the Brazil - French Cooperation for Fast Reactor technology development.

In 1970 the physicist Roberto Stasiulevicius who had recently arrived from IPEN-CNEN-SP constructed a neutron spectrometer in IEN, a wood prototype with a simple goniometer with the collaboration of the COPPE-UFRJ students Saturnino F.Mauro and Hélida L. do Nascimento.[9]. This instrument was substituted later by a new crystal spectrometer (EC-IEN) designed and constructed by the engineer Luiz Arthur Bezerra França with the collaboration of the mechanical technician Antonio J.M. Rebello.

Some time after this, the researcher Roberto Stasiulevicius (RS) initiated measurements of the total neutron cross-sections for some chemical elements such as Au, Ag, and Cd to help determine the thermal neutron spectrum emerging from the J-9 irradiation channel, the more intense beam hole of the Argonaut reactor.

In 1975 researchers from IPEN, A. M. Figueiredo Neto, Roberto Fulfaro e Laércio Antonio Vinhas, were invited to cooperate in measurements of the thermal neutron spectrum of the Argonauta reactor and they joined IEN researchers to use the EC-IEN operating with an aluminum single crystal in transmission [10,11,12].

In 1976 the physicist Dante Luiz Voi initiated his post-graduate thesis using the facilities of IEN and implemented some improvements in the crystal neutron spectrometer under the orientation of Dr John Douglas Rogers from COPPE-UFRJ and the researcher Roberto Stasiulevicius (RS) from IEN. The instrument was used for total neutron cross-section determination of the first resonance of the rare earth Lu used as a neutron thermometer in evaluating the thermal spectrum of reactors [13].

In 1979 the latter was transferred to the Instituto de Pesquisas Radiotivas(IPR-CNEN-MG) and the former continued working in the EC-IEN adding accessories that allowed applications of it in the Argonauta reactor spectra [14] and neutron cross-section determinations to the control of purity of materials, used in structural components of reactors, such as. graphite, aluminum, zircalloy, steel, light and heavy water.

In the 80s as part of the Brazilian project for the Reator Produtor de Radisótopos (RPR) in IEN-CNEN-RJ, pellets were designed containing uranium, graphite and bakelite that made up part of the reactor fuel element. Manufacturing control for this new fuel was necessary and opened an extensive field of research including measurements of total neutron cross-sections of the bakelite (as a new moderator)[15,16] with the EC-IEN and the chopper with time-of-flight/Be filter spectrometer of IPEN-CNEN-SP. This work was developed with the special collaboration of Dr. José Mestnik Filho from the latter institution and resulted in the doctoral thesis [17] of the researcher Dante Luiz Voi from IEN. In this work, an as yet unpublished form was developed for control of the composition and formulae of chemical compounds by the grouping and parceling neutron cross-sections method developed in IEN. Much neutron data obtained using the EC-IEN were used, together with that from the Barn Book and international publications as well as data obtained previously by Drs. Lia Queiroz do Amaral, Silvio Bruni Herdade, Laércio Antonio Vinhas, Roberto Fulfaro, José Mestnik Filho and others using the IPEN spectrometers.

In the 90s the government initiated a program of re-organization and transformed all positions within the divisions of the state institutions into jobs, requesting the return of funds by researchers and requiring that applications should be in healthcare and nutrition, areas with some of the challenges that Brazilian society face. It was the first step for involvement of the IEN reactor division with the medical area and an initial project was developed with the cooperation of the physician Hélio Rocha from the pediatric institute of the Universidade Federal do Rio de Janeiro (UFRJ). In the first work, H and D total neutron cross-sections measurements were used for the development of a quantitative method for determination of body water in subjects [22].

Following these applications some general publications were made about spectrometry and diffraction in Brazil [23,24] and as a millennium program in the IEN was initiated an amino acids project of neutron cross-sections measurements. A new collaborator, the electronic technician Francisco José Ferreira joined to the neutron spectrometry group helping in the operation of the machines, Argonaut reactor and EC-IEN. Studies were carried out for applications of neutron spectrometry in the control of purity of amino acids used as administration in hospital patients [25]. Twenty essential and non-essential amino acids were studied. Later in 2009 a small neutron data file was prepared in IEN reporting the measured total neutron cross-sections of these amino acids, of many compounds and data for which also were collected from bibliographies. All this data is used for manipulation in the grouping and parceling method previously reported. The amino acids are important in the construction of largest molecules as enzymes and proteins and some of them simulate human tissues for applications in dosimetry. Because of the amino acids composition, mainly of H and C, neutron spectrometry is a good technique for their control.

At the end of this work is presented the principal bibliography but the original paper to be published in Portuguese [30] reports the complete and more detailed version of the historical facts and the complete bibliography with a large number of citations and this can be disseminated to everyone interested.

3. THE OLD AND NEW NEUTRON DIFFRACTOMETERS, THE CHOPPER TIME-OF-FLIGHT/BE FILTER AND THE TRIPLE AXIS SPECTROMETER OF IPEN-CNEN-SP.

The old neutron diffractometer was designed and constructed for multiple purpose applications and used a flat mosaic Cu(200) monochromator at 36° take-off angle and Soller collimators 20´, 27´ and 71´ horizontal divergences for the in-pile, incident-beam and scattered-beam collimators, respectively and a boron tri fluoride detector. This system was implemented with the collaboration of Dr. Carlos Benedito Ramos Parente, currently physicist and researcher at IPEN and two researchers from the AIEA, Norris Nereson and Robert G. Wenzel also with the collaboration of the USP physics students Kouji Harada and Yukiu Koishi.

In 1972, a triple axis neutron spectrometer (TAS) was constructed that allows measurement of the scattering function at any point in energy and momentum space. Another important instrument constructed at the same time was the chopper with time-of-flight and Be filter spectrometer that enabled measurements of frequency spectra of polycrystalline and amorphous materials to be made for double differential cross-sections determinations resulting in many national and international publications.

In 2005, a new diffractometer (PSD) [26] was designed for powder studies with the monochromator-to-sample distance reduced from 1.5 to 1.17 m, compared to the old diffractometer. A sample of 5 mm diameter and 5 cm height was kept. The detector for PSD formed by 9 linear detectors 60 cm long was placed 1.07 m from the sample, in the new configuration. The sample-to-detector distance results in a 30° span for the PSD and a 4 mm spatial resolution was assumed, resulting in 150 intensity points measured simultaneously. Computed intensities at the detector represent sums over electronic channels corresponding to the PSD spatial resolution. The gain in rate of data collection was increased and a 61° take-off angle was firstly assumed for the new configuration. At this angle, only the Si(311) reflection is of real interest because it produces neutrons of 1.66 Å which is convenient for stress scanning. The take-off angle was then increased to 84°. The main advantage of this angle is in the multiple choice of wavelengths. Four different wavelengths can be obtained with a simple rotation of the monochromator (one of them needs an additional tilt). The new instrument has a better resolution and is around 600 times faster in data acquisition, when compared with the old one.

The PSD neutron diffractometer (DN PSD-IPEN) of IPEN-CNEN-SP was designed mainly for crystalline and magnetic structure determination and application of the Rietveld method in multiphase analysis. However, under request, other types of application can be considered. Utilization of this new instrument will be open to the Brazilian and Latin American scientific and technological communities.

4. THE OLD AND NEW CRYSTAL NEUTRON SPECTROMETER OF IEN-CNEN-RJ

The old crystal neutron spectrometer was a wood prototype with a simple goniometer, a boron tri fluoride detector, operated with lithium fluoride crystals and was installed in J-9 beam hole of the Argonauta reactor. The collimators were constructed with walls of borated paraffin, has low angular resolution and high fast neutron and gamma radiation background. It was substituted some time after by a new crystal spectrometer with a milling machine table and an imported precision goniometer of 6 degree of freedom that operates with a natural calcite crystal.

The new crystal neutron spectrometer (EC-IEN) has two reflected Soller slit collimators with angular divergence of 50'. A calcite crystal with Fankuchen cut gives a Gauss form rocking curve with one degree at FWHM. Fast neutron and gamma radiation background are suppressed by Cd plate and borated paraffin in the beam hole surrounding area. The versatility of the system for moving components in the J-9 irradiation channel allows the intensity of the neutron beam to be increased or diminish, controlled by neutron beam monitor records. A small cylinder of graphite with a rough surface is placed near the reactor core to work as a neutron trap to increase the intensity of the 1 inch collimated neutron beam. This and other alterations in the beam hole resulted in an increase by a factor of four in the neutron beam intensity as compared with the oldest configurations.

5. FUTURE DEVELOPMENTS IN NEUTRON SPECTROMETRY AND DIFFRACTION.

A realistic example of future applications for neutron diffraction is in stress analysis in aerospace and automotive components. It can also be employed in 3D structure studies of molecules or molecular groups such as nanotubes, fullerenes, etc.

In medicine and related areas neutron spectrometry and diffraction can be used to establish the dynamics and structure of materials containing low atomic numbers elements like amino acids, proteins and other components of live cell and of the unidentified internal structures of DNA because these materials have high neutron cross-sections.

A small angle neutron scattering (SANS) spectrometer was designed and is in study to be constructed in IPEN-CNEN-SP[27]. It will open an interesting and important field of neutron diffractometry and espectrometry related applications .

In 2010, a cold neutron source was designed at IEN-CNEN-RJ [28] to be installed near the core of the Argonauta reactor as a prototype of a source to be installed in the Reator Multipropósito Brasileiro (RMB). This system would enable an increase to be achieved in applications for neutron spectrometry, tomography, neutrongraphy and other irradiations. The reactor's construction will depend on the government decision whether or not to carry on with the project.

In IPR reactor at CDTN-CNEN-MG, a vertical neutron beam extractor provides good intensity of radiation for neutrongraphy with possibilities to be installed around a neutron spectrometer. One of the last projects and a dream of Dr Roberto Stasiulevicius for the IPR reactor was construct a horizontal neutron beam extractor crossing the underground and furnishing neutrons for many arrangements into a room away and at the same level of the IPR reactor core.

6. CONCLUSIONS

Nowadays the international scene of neutron applications is enormous having as reference the High Flux Reactor(HFR) of the ILL(Institute Laue-Langevin) in Grenoble-French having many experimental arrangement around it. Neutron spectrometry and diffraction, a new horizontal-surface reflectometer and the small-angle neutron scattering instruments is playing an essential role in many field of the sciences,. however Biology has consolidated its position as a mainstream part of the researches.

At ILL 2020 Vision Meeting in Grenoble on September 2010[29], neutron scattering was presented and discussed as the more important work area and pointed to soft condensed matter science applications. In magnetism and superconductivity fields for example, the last generation superconductors pnictides and chalcogenides continue to elude a definitive theory of the mechanism of the effect and is a subject for the next decade.

In Brazil the small number but selected instruments of neutron spectrometry and diffraction linked with other techniques such as X-ray diffraction for example, allowing an extensive field of cooperation and producing realistic results to be applied to solving problems that are faced mainly by society. However, it is necessary to ensure continuous support for researches to profit from these fifty years both in terms of the knowledge acquired and the trained personnel who are still on the scene in our institutions.

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