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SYNCHROTRON RADIATION (SR) IN MATERIALS SCIENCE: A BRIEF RETROSPECTIVE ON FIRST APPROACHES TOWARDS THE USE OF SR AT EUROPEAN RESEARCH FACILITIES

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ABSTRACT: A brief quasi-historical survey is presented on the primary joint efforts developed by Portuguese researchers towards the use of synchrotron radiation at European Instrumental Facilities, emphasizing some original contributions and mentioning a few decisive steps towards the adhesion of Portugal to the European Synchrotron Research Facility (ESRF, Grenoble/France).

Keywords: Materials science; minerals; synchrotron radiation; ESRF.

1. DISCLOSING THE USE OF SYNCHROTRON RADIATION

Twenty five years ago, a workshop on X-ray spectroscopy in atomic and solid state physics was organized at Vimeiro under the auspices of the NATO Advanced Study Institute (August 30 to September 2, 1987). The organizers were Profs. José Gomes Ferreira and Maria Teresa Ramos from the Physics Department of the Faculty of Sciences, Lisbon University [1].

Portuguese researchers working in the fields of Physics, Chemistry, Biology, Geology and Materials Science, had then the opportunity to get in touch with European Scientists devoted to the use of synchrotron radiation instrumental facilities in a wide range of applications – namely, the past Dutch Prof. John C. Fuggle from the Catholic University of Nijmegen/ Netherlands, working currently at BESSY I [*Berliner Elektronenspeicherring-Gesellschaft für Synchrotronstrahlung m.b.H.*] (figs. 1 and 2), and the French scientists Pierre Chevallier, Alain Fontaine, Pierre Lagarde and Robert Cortes from the LURE [*Laboratoire pour l'Utilization du Rayonnement Electromagnétique*] in Orsay (figs. 3 and 4).

2. EARLY PRELIMINARY CONTRIBUTIONS

Having for a long time dealt with X-ray diffraction and fluorescence spectrometry, and with the aim of performing spectroscopy tests for light elements by using just a normal analytical wavelength dispersive X-ray fluorescence spectrometer, a contribution was presented on sensitivity of $K\beta$ emission lines to the chemical bonding of aluminium, silicon, phosphorous and sulphur [2] in minerals using a current laboratory wavelength dispersive X-ray fluorescence spectrometer.

In the sequence of this presentation at the Vimeiro workshop, Prof. J. Fuggle invited the authors to join the research group working at BESSY I on low energy X-ray absorption spectroscopy (XAS) under his supervision. Along the following decade, a Ph.D. thesis came out from this collaboration and ten papers were published dealing with XAS in total-electron yield (TEY) and fluorescence yield (FY) modes.

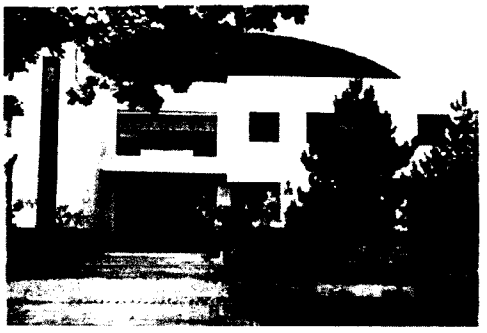

Fig. 1. Photo of the former BESSY I in the Lentzeallee / Berlin (dated 1991).

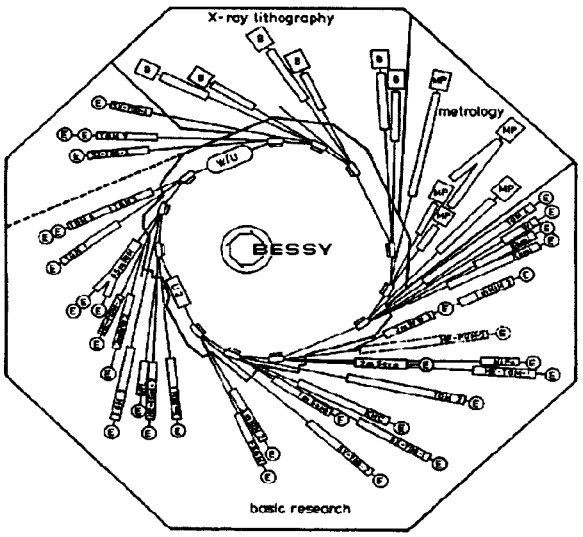

Fig. 2. Sketch of the beam-lines available at BESSY I synchrotron in the late eighties / early nineties.

Low energy edges were then studied at BESSY, namely, the *L*-edges of *3d* transition metals in various compounds, both natural (e.g., titanium in oxide minerals [3]) and synthetics (e.g., titanium in ceramic composites [4], manganese and iron in chromium oxide films [5]).

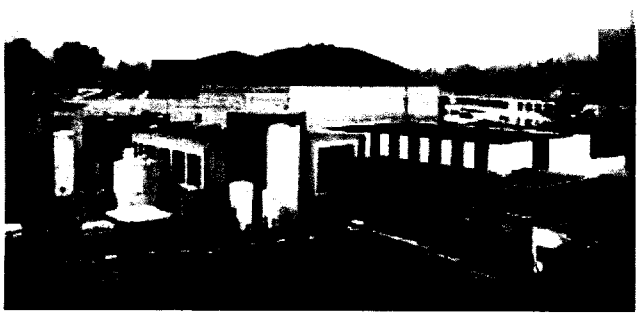

Fig. 3. The former DCI Synchrotron at the LURE in Orsay / France.

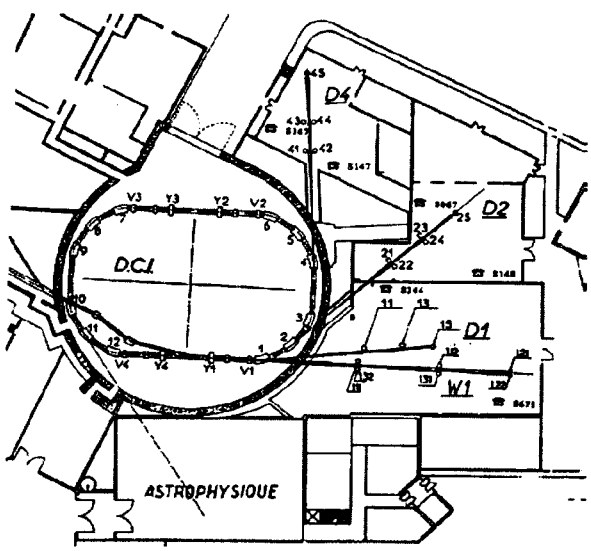

Fig. 4. Schematic diagram of the DCI synchrotron and beam-lines there available. The most looked for by the Portuguese users were D-15A for micro-chemical characterization applying X-ray fluorescence spectrometry (μ–XRF-EDS) and D-21 plus D-13 for studies based on X-ray absorption spectroscopy.

The study of the oxygen *K*-edge in oxide minerals deserved particular attention [6].

At the same time, a close collaboration was implemented with Scientists from the LURE. Along fifteen years various researchers took to the DCI synchrotron samples of minerals and other materials for elemental analysis using the photon microprobe developed by P. Chevallier, addressing particularly the analysis of heavy elements present as traces (e.g. [7-10]). Studies based on X-ray absorption spectroscopy were also performed (e.g. [11]).

3. ...AND PORTUGAL JOINED THE ESRF !

Ten years after the Vimeiro NATO-ASI workshop, Portugal joined the ESRF as an Associated Scientific Member (ACI) following a proposal submitted to the Ministry of Science and Education, Prof. Mariano Gago, by a group of researchers working in the domains of Physics, Chemistry, Biology and Materials Science:

- Mª Teresa Ramos (Dept. Phys., Fac. Sci., University of Lisbon)
- Mª Arménia Carrondo (ITQB / Inst. Chem. Technol. Biochem., New Univ. of Lisbon)
- Mª Margarida Costa (Dept. Phys., Fac. Sci., University of Coimbra)
- Mª Ondina Figueiredo (Mater. Sci. Dept., Fac. Sci. Technol., New University of Lisbon)

The subscribers of that proposal had formerly developed successive efforts in the sense supporting fundaments to this adhesion, as exemplified by [12].

According to data from the Portuguese Foundation for Science and Technology [13], the contract between Portugal and the ESRF has implied a contribution of ~1% to the total budget of this European Laboratory. This partnership agreement was renegotiated in 2008 for the next five years.

Since 2005, a mean of about forty research teams from Portugal profit annually from beam-time allowance to perform experiments at the European synchrotron in Grenoble. Nowadays, Portuguese research teams have access to a large group of synchrotron radiation facilities with the support of EU, thus enlarging significantly the opportunities to develop a wide and challenging variety of scientific experiments applying this powerful "light".

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