CENTRAL LABORATORY OF X-RAY AND ELECTRON MICROSCOPY RESEARCH AT THE INSTITUTE OF PHYSICS OF THE POLISH ACADEMY OF SCIENCES, WARSAW [1*)]

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The beginning and history of the Central Laboratory of X-ray and Electron Microscopy at the Institute of Physics of the Polish Academy of Sciences in Warsaw is described. Then, recent scientific achievements are presented. Organising activities of the Laboratory staff are also mentioned.

1. History

The Central Laboratory of X-ray and Electron Microscopy Research at the Institute of Physics of the Polsh Academy of Sciences in Warsaw has a long history. In 2008 it celebrates a jubilee of 35th anniversary. In fact, a background is much longer because the crystal structure research was carried out at the University of Warsaw by a group headed by Professor Stefan Pieńkowski. In 1953 the Institute of Physics of the Polish Academy of Sciences was founded with departments located not only in Warsaw, but also in Wrocław, Poznań, Kraków, and Toruń [1]. Prof. Pieńkowski was appointed to a director of the Institute and Prof. Leopold Infeld to a chairman of the Scientific Council. The development of the Institute was fast. As a continuation of the work at the University of Warsaw, in 1966 the Department for X-ray Physics was organised in Warsaw, and Prof. Julian Auleytner was established as its leader.

In 1973 the Institute of Physics was reorganised. In Warsaw the research topics were concentrated in three scientific divisions and two central laboratories. The Department of X-ray Physics was turned into the Central Laboratory of X-ray and Electron Microscopy, created for a double purpose: scientific investigations concerning a development of material characterisation, and scientific expertises for other groups of the Institute and for other research institutions as well as of medicine and industry.

Prof. Julian Auleytner took up a leadership of the Laboratory and he was its head for twenty years. In the period of 1993-1999 Prof. Tadeusz Figielski was a head of the Laboratory. Since 2000 Prof. Krystyna Jabłońska is its leader.

Initially, the Laboratory included three scientific groups: the Group for Real Structure Research and X-ray Spectroscopy, the Group of Physics of Defects in Semiconductors, and the Group of Electron Microscopy, as well as the Technical Support Group. At that time the Laboratory was equipped with modern X-ray and electron analytical tools. From the beginning the Laboratory has collaborated with many foreign scientific groups.

In 1986 The Group of Applied Crystallography was established. Next, the Technical Support Group was closed down, whereas the Group of X-ray Fluorescence and Electron Microprobe was created which was then transformed into the Group of X-ray Spectroscopy and Microanalysis. In late nineties the Group of Secondary Ion Mass Spectrometry was founded. In 2000 the Group of Physics of Defects in Semiconductors moved to the Scientific Division of Physics of Semiconductors and simultateously the Group of X-ray Optics was established which in 2004 joined the Group for Real Structure Research, creating together the Group of X-ray Optics and Atomic Structure Research. At the same time the Group of SIMS was taken in by the Group of X-ray Spectroscopy and Microanalysis and the Group of Biological Physics was founded.

At present, in 2008, the Laboratory consists of five scientific groups: the Group of X-ray Optics and Atomic Structure Research, the Group of X-ray Spectroscopy and Microanalysis, the Group of Applied Crystallography, the Group of Electron Microscopy, and the Group of Biological Physics.

[1 *)] In this issue we begin a presentation of Polish research groups involved in synchrotron radiation investigations and workers of which are active members of the Polish Synchrotron Radiation Society. We start with the Central Laboratory of X-Ray and Electron Microscopy Research at the Institute of Physics of the Polish Academy of Sciences in Warsaw.

Figure 1. Laboratory staff and visitors gathered at the Institute entrance during the celebration of the 30th anniversary in 2003

As the result of the 35 year-scientific activity of the Laboratory, over 1500 papers were published in the international scientific journals and 28 research workers received the PhD degree. At present, five PhD students are preparing their theses at the Laboratory.

Several persons of the staff went abroad, and actually they work at the scientific institutions in Federal Republic of Germany, the USA, Canada, and Australia. Two young researchers are holders of post-doc scholarships in the USA and Germany.

The researches of the Laboratory were winners of several prizes [1]. These of the greatest prestige were two international awards: in 1976 the Polish Academy of Sciences and Academy of Sciences of German Democratic Republic awarded an international prize to Prof. J. Auleytner and Prof. J. Heydenreich, and their co-workers for development of X-ray and optical method for solid state physics and in 1985 the Scientific Secretaries of the Polish Academy of Sciences and Bulgarian Academy of Sciences to Prof. J. Auleytner and Prof. G. Grigorov, and their coworkers for investigations of adsorption properties of metallic surfaces. Five times members of the staff were given prizes of the Scientific Secretary of the PAS: in 1972 the group directed by Prof. J. Auleytner, in 1979 the group headed by Prof. T. Figielski, in 1984 the group of Dr. G. Jasiołek, in 1988 Dr E. Sobczak and Prof. J. Auleytner, and in 1989 the group directed by Dr J. Bąk-Misiuk in co-operation with the Institute of Electronic Materials Technology.

The establishment in 2002 the Centre of Excellence CEPHEUS (Centre of Photon, Electron and Ion Advanced Methods for Natural Science) with the European Commission support for 3 years under the direction of Prof. K. Jabłońska, the leader of the Laboratory, was also a great achievement.

2. Equipment

Studies of the solid state require a good technology for preparing samples (crystals, thin films, low dimantional objects) and a research equipment for a characterisation of them as well as an understanding their crystal and defect structure. At early years the structures were characterised using commercial cameras, diffractometers, and spectrometers. In the Department of X-ray Physics, self-made cameras and high resolution diffractometers were built, among them an X-ray camera with oscillating crystal and film [2] and a moving slit for X-ray automonochromatization [3], and used here and at other institutes.

After the creation of the the Central Laboratory of X-ray and Electron Microscopy, at early seventies of the

20th century, it received respectable amount of money for purchase of several modern pieces of apparatus: an electron probe micro-analyser JXA-50A with scanning electron microscope and X-ray energy dispersive spectrometer LINK (JOEL), a double axis X-ray diffractometer of Bond type with temperature chamber, equipped with a four crystal monochromator of Bartels type, an X-ray powder diffractometer with secondary monochromator for high sensitivity phase analysis (SIEMENS Kristalloflex 4 made in Germany), a soft X-ray grating spectrometer RSM-500 (made in USSR), an electron diffractometer (RHEED, TED) ENR-102 (made in USSR), and a metallographic microscope Reichert MeF2. Several devices were projected and constructed by the staff of the Laboratory: an automated diffractometer for powders and single crystals, an automated diffractometer for powders, and a bremsstrahlung isochromat spectrometer.

After a long time of use these devices became old and inadequate to nowadays tasks of the Laboratory. Therefore the leaders and staff made great efforts to upgrate an equipment. In late eighties a high-resolution electron transmission microscope JEM 2000 EX (JEOL) was bought for the finest structure studies at the nano- and atomic scale. In nineties the Laboratory was equipped in various facilities permiting for the structure and composition determination, in particular with two modern high-resolution diffractometers Philips MRD and X'Pert MPD and with powder diffractometer/ reflectometer X'Pert MRD with semiconductor linear position-sensitive detector as well as a secondary ion mass spectrometer IMS 6f (CAMECA, France) for an analysis of atomic composition of materials, determination of mass spectra and element's depth profiles.

At present a variety of materials are studied, *e.g.* single crystals grown by the Bridgman, Czochralski, chemical transport and vapour methods and thin layers and multilayers prepared by MBE, MOCVD and sputtering methods as well as minerals and biomedical materials. Nowadays, the experimental methods at laboratory are either completed or replaced by methods provided at the synchrotron beamlines. Starting from early nineties, the scientists of the Laboratory usually get more than ten weeks of beam-time at the experimental stations of various synchrotrons, what helps to solve various scientific tasks. Many research projects have been performed for several years in co-operation with the European and American laboratories owing the national synchrotron radiation sources.

3. Recent scientific activities

The research activity of the Laboratory has an interdisciplinary approach and concerns the comprehensive characterisation of matter. Over the past years, numerous techniques were developed which determine, on an atomic scale, the structure of the matter and which enable the physicists to describe, with increasing precision, basic interactions between the component atoms.

The recent scientific activity of **the Group of X-ray Optics and Atomic Structure Research** is focused on investigations of an interaction of intense extreme ultraviolet (XUV) and X-ray synchrotron beams with solids. In particular, damage processes induced on solid surfaces with XUV-FEL femtosecond pulses are studied, as well as resistance to the damage for the materials applicable in the optical components for the new radiation sources is determined. Experimental studies are compared to simulations of the propagation in solids of strong electromagnetic pulsed beams within the XUV and soft X-ray radiation (SXR) wavelength. The propagation models are computed in function of the intensity and pulse duration in the ranges up to 10^{14} W/cm^2, and down to 30 fs, respectively [4, 5]. A significant part of experimental results has been achieved with a unique experimental station FELIS (Free Electron Laser - Interaction with Solids) designed and constructed by the scientists and engineering staff of the group to study interactions of intense femtosecond vacuum ultaviolet (VUV) pulses with matter at TTF1 Free Electron Laser in Hamburg [6]. Apart the XUV-FEL directly related work, surface modifications and defect structures induced by other damage processes, such as ion implantation, or ablation with picoseconds pulses generated by optical lasers are also investigated [7]. The surface modifications are studied with a variety of techniques, that includes optical interference-polarization microscopy, RHEED, X-ray scattering methods with synchrotron radiation, raman spectroscopy, and the others. The above mentioned main scientific interests of the group are supplemented with structural research of low dimensional structures and nanomaterials by means of methods employing intense X-ray beams of synchrotron radiation. An example here can be a determination of structure and electrical properties of thin films composed of gold nanoparticles spaced by dithiols of various length [8].

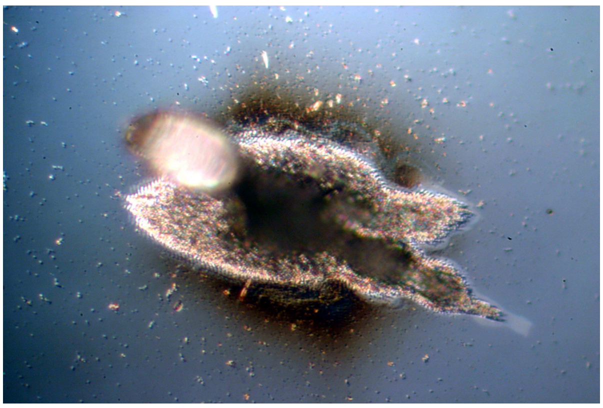

Figure 2. Ablation crater created on Si surface by a few shots of femtosecond free-electron laser pulses (interference-polarizing microscope with Nomarski contrast).

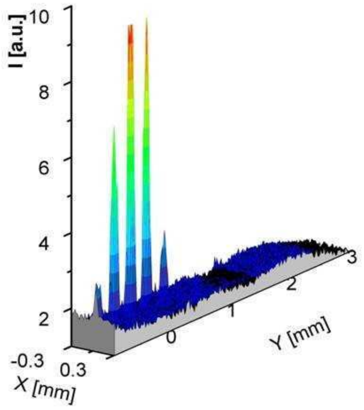

Figure 3. X-ray diffraction map showing strain distribution around points on Si (001) surface irradiated by a few FEL pulses. The map was obtained at BM-20 beamline at the ESRF, Grenoble.

The activity of **the Group of X-ray Spectroscopy and Microanalysis** is devoted to charactersation of the variety of materials independently of their states (liquid, glasses, amorphous or crystals). The techniques used for this purpose are X-ray absorption spectroscopy (XAS), electron probe microanalysis (EPMA), and secondary ions mass spectroscopy (SIMS). By means of these methods the content of given element in the materials is estimated, mapping element distribution and depth profile are determined. Exploiting the advanced analysis of XAS the local atomic structure around elements, as well as their chemical bonds and ionic state is determined. Analysis of the EXAFS oscillations is a source of information on a short-range order in the samples. This is of particular value in the case of investigation of buried low dimensional structures or dopants in semiconductors. The example of application of this technique to study of strains and Si concentration inside Ge quantum dots formed in silicon is presented in the paper [9] and the location of Mn implanted into Si crystals in the paper [10]. Due to the fact, that the shape of the XANES spectra depends on the density of the unoccupied states in a given compound, XANES can be used for testing the solid state theory applied for estimation of other physical properties of matter. The examples of this kind of comprehensive studies were published for III-V family of semiconductors [11]. Recent interests of the group is devoted to the biomaterials. The local atomic structure of di-alanine amino acid derivative of protoporphyrin IX used in the photodynamic diagnostic and therapy of cancers was investigated to find the location of Fe atoms [12]. The location of Fe in the chitosans was found using complementary XAS and magnetic studies [13]. Chitosan and its derivatives have a variety of current and potential applications e.g. in biomedical products, cosmetics, food processing and removal of metallic impurities from wastewaters.

For phase analysis, crystal structure refinement of polycrystals, and for understanding the defect structure of bulk crystals, thin films and multilayered samples, **the Group of Applied Crystallography** uses various high resolution and powder X-ray diffraction as well as scattering methods. A large part of studies are performed under extreme conditions (low/high temperature, high pressure) or after a high-pressure-high-temperature treatment. The scientific activity of the group includes the determination of the thermal expansion and compressibility for semiconductor materials, determination of the influence of carriers, dopant atoms and defects on the lattice parameters in semiconductor thin films. Examples of the activities concern the structure refinement using the Rietveld method [14], thermal expansion for spinel-type silicon nitride [15] (Fig. 4), multiple diffraction effect in strained thin ZnSe film [16] (Fig. 5). Recently the group is also involved in the studies of strained GaAs thin films [17]. Moreover, the structure of potential spintronics materials, *e.g.* diluted ferromagnetic GaMnAs semiconductor, GaAs films with embedded MnAs magnetic nanoclusters as well as silicon implanted with Mn is investigated. In particular, small precipitates within the Si:Mn near-surface region as well as changes in the lattice after introduction of magnetic inclusions have been characterised [18].

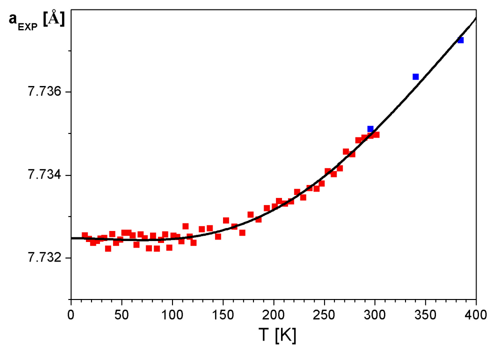

Figure 4. Temperature dependence of the lattice parameter of spinel-type Si_3N_4 [14]. The low temperature data are obtained at B2 beamline (Hasylab).

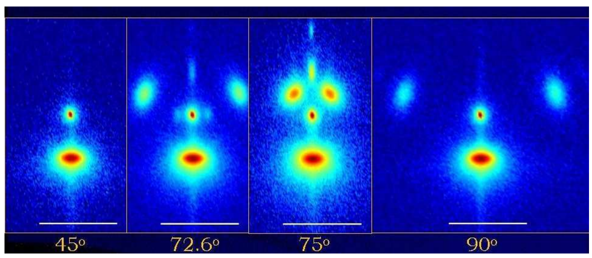

Figure 5: Reciprocal space maps for 002 reflection of ZnSe (1 μm thick) relaxed layer on GaAs (001), for various azimuthal angles, φ = 45, 72, 75 and 90, of the sample [15]. Additional spots are observed in the vicinity of the main reciprocal lattice point, due to the multiple diffraction effect. The azimuthal angle related to [100] direction on the surface is indicated below each image. Analysis of such effects permits for analysis of the strain state of the layer.

The Group of Electron Microscopy is involved in the structure characterisation of semiconductors, metaloxides, supercoductors and fullerenes nano-objects. Computer aid methods of analysis and simulation of diffraction patterns and transmission electron microscopy images are developed and applied to solve the structure of new materials [19, 20] and to a quantitative determination of strain fields and chemical composition at atomic level in semiconductors' heterostructures [21].

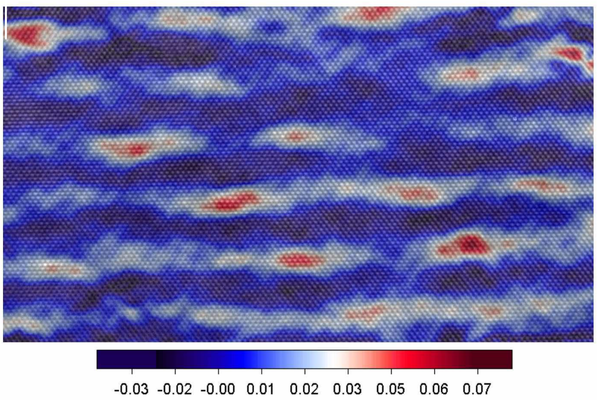

Figure 6. Cross-sectional HRTEM image in 110-zone axis of stacked ZnTe/CdTe QDs structures. Colors illustrate the relative local lattice parameter values measured from the image of CdTe/ZnTe superlattices.

The youngest group, **the Group of Biological Physics** participates in interdisciplinary studies, experimental and theoretical, which involve physics, biology, chemistry, and bioinformatics. One domain of the research is focused on molecular mechanisms of regulation of eukaryotic protein biosynthesis and mRNA turnover [22, 23], in particular, structure - function relationships for the specific ligands of the proteins involved in these processes and also thermodynamic aspects of intermolecular recognition. The main experimental techniques used are: emission spectrosopy, surface plasmon resonance, and atomic force microscopy. The members of the group work also on toxicity mechanisms of the β-amyloids in the context of the Alzheimer disease [24] as well as they study ways to generate optimal biodegradable drug transporters and diagnostic sensors that could enter live biological cells. The leader of the group, Marek Cieplak, uses computer simulations to elucidate mechanisms concerning single protein manipulations (such as stretching by using the atomic force microscope) [25], protein folding, and effects of confinement and hydrodynamic interactions on proteins. He is also involved in providing biological interpretation of data obtained by using the genetic micoarrays on the genetic activity of all genes in an organisms such as yeast [26].

4. Organising activities

From the very beginning, the staff of the Laboratory was very active in the organisation of national and international meetings concerning the variety of X-ray and electrons based methods. During the period of thirty five years of the activity one Congress, two series of international conferences, and one series of national symposia were organised. In 1978 the XIth International Congress on Crystallography for 1650 participants was organised in Warsaw by the staff of the Laboratory and the Institute for Low Temperature and Structure Research of the PAS, Wroclaw. The Congress was the great success of Polish organisers. In the period 1964-1992 the Laboratory organised eight *International Schools and Symposia on Defects in Crystals*. Due to these schools the Polish scientists had an opportunity to be in touch with colleagues from the West and East. It was very important at that time when the distribution of scientific information was hidebound.

Recognising the importance of synchrotron radiation for X-ray physicists, Laboratory decides to disseminate actively the knowledge about these new sources of radiation as well as the scientific achievements among the scientific community in Poland and Central and Eastern Europe (CEE). In 1991 the Polish Synchrotron Radiation Society (PSRS) was created by the scientists of the Laboratory together with researchers of other scientific institutions in Poland with the main goal to organise national and international synchrotron radiation schools. Till now, nine *International Schools and Symposia on Synchrotron Radiation in Natural Science* and seven *National Symposia of Synchrotron Radiation Users* were organised. The Proceedings of these meetings were published in *Acta Physica Polonica A* and *J. Alloys and Compounds*. Scientific workers of the Laboratory have been the chairmen and main organisers of symposia of European Materails Research Society Fall Meetings in Warsaw since 2003, and many other meetings and workshops, among them *East European Meeting on Synchrotron Radiation and Free Electron Laser Sources* in Kraków-Przegorzały in 1999*, Workshop on Extended X-ray Absorption Fine Structure Analysis* in Warsaw in 2001, *Workshop on New Methods of Low-Dimensional Structures Characterisation: VUV and X-ray Free Electron Lasers* in Warsaw in 2002*, Workshop on Advanced Methods for Interpretation of TEM, X-ray and SIMS Measurements in Nano and Atomic Scale* in Warsaw in 2005, and *Workshop on Application of X-ray Absorption for Determination the Local and Electron Structure of Materials* in Warsaw in 2006.

The scientists of the Laboratory are on the go in the Polish Synchrotron Radiation Society; they promote and actively participate in works of the National Centre of Synchrotron Radiation in Cracow as well as support an idea of the construction of a Polish free electron laser POLFEL in Świerk. The Polish access to the European Synchrotron Radiation Facility in Grenoble is realised due to a special grant coordinated by Prof. Jabłońska, the President of the PSRS and the head of the Laboratory.

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