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National Institute for Materials Physics
Bucharest-Magurele
(NIMP)

launches

THE EURO-REGIONAL CENTRE FOR THE STUDY OF ADVANCED MATERIALS, SURFACES AND INTERFACES

POS-CCE Project
„Euro-Regional Centre for Studies
of
Advanced Materials, Surfaces and Interfaces“
(CEUREMAVSU, SMIS-CSNR code 2665)

1. GENERAL DATA

This Centre, whose acronym is CEUREMAVSU (code SMIS-CSNR 2665), could be developed due to funding obtained through the Sectorial Operational Program – Increase of Economic Competitiveness (POS-CCE), Priority Axis 2: Research, Technological Development and Innovation for Competitiveness, Operation 2.2.1 Development of Existing R&D Infrastructure and Establishment of New R&D Infrastructures (laboratories, research centres). The center consists of two newly established and five modernized laboratories. As part of the project, 23 pieces of equipment worth over 100 000€ each were acquired and 24 new positions were created for highly qualified specialists (physicists, chemists, engineers).

1.1. Funding

The total value of this project was 43 004 595lei, of which the sum contributed by NIMP represented 20 367lei. The European Union contributed 32 968 903lei, and the Romanian Government 10 015 325lei.

1.2. Results Indicators

By contract, it was established that the main indicator would be the number of pieces of equipment worth more than 100 000€ for 18 (eighteen) pieces of equipment. After the acquisition procedures (auctions, negociations), the prices obtained in the end for the great majority of the equipment were lower than the initial estimations, which allowed the acquisition of 23 (twenty three) pieces of equipment worth more than 100 000€ each. The need for the additional equipment was decided based on a reevaluation of the Feasibility Study, which determined that this equipment will contribute decisively in placing NIMP in a top position for materials physics and solid state research at the European level.

Another important indicator of the Project was the number of positions created within 5 years after the start of the project (and 3 years after the end of the project). This number of positions was established at 24 (twenty four). At the moment the project was drawn to a close (two years after its beginning), these positions were created and occupied by young graduates in the fields of physics, chemistry or engineering.

It can be concluded that, at this moment, the two major indicators of the Project have already been surpassed. The other results indicators (number of software packages, number of ISI papers produced, positions maintained for R&D activities, valuable postdoctoral researchers attracted etc.) have been realized adequately as well.

23 pieces of equipment acquired are worth over 100,000€ each:

1. Ferroelectrics Tester TF 2000 E (Aix ACCT)
2. Angle- and Spin-resolved Photoelectron Spectroscopy (Specs)
3. Raman Microscope (Jobin Yvon)
4. Near Field Optical Microscope (ABL Jasco)
5. Cryoprober (LakeShore)
6. Puls and Fourier-transform Electron Spin Resonance Spectrometer (Bruker)
7. Vector Network Analyzer (Agilent)
8. High-Resolution Transmission Electron Microscope (Jeol)
9. FIB-SEM focused ion beam sample preparation system (Tescan)
10. Mössbauer Spectrometer with magnetic field, ultralow temperature cryostat (Englemann Scientific)
11. PPMS system for physical properties measurements (Quantum Design)
12. SQUID superconducting quantum interference magnetometer (Quantum Design)
13. Two He Liquefaction Machines (Cryomech)
14. Design and Execution of Class ISO 1000 and 100 Cleanroom Assembly (EDAS EXIM)
15. LEEM-PEEM Low energy and Photoelectron Electron Microscope (Specs)
16. Scanning electron microscope and nanolithography system (Raith, Hitachi)
17. SPM Scanning Microscopy Station (NT-MDT)
18. Small dimension line measurement station (LakeShore)
19. Photolithography System (EV Group)
20. Microwave Spectrometer up to 7 THz (Aispec)
21. Metallization system for deposition of noncontaminated metals (Bestec)
22. Metallization system for deposition of contaminated metals (Bestec)
23. XAS X-ray Absorption Spectrometer (Rigaku)

2. DESCRIPTION OF THE EURO-REGIONAL CENTRE FOR THE STUDY OF ADVANCED MATERIALS, SURFACES AND INTERFACES

2.1. The Centre has planned to establish two new laboratories:

A1. The High-resolution Transmission Electron Microscopy Laboratory
In this laboratory, the main acquisition was **(i)** a Jeol ARM 200F high resolution electron microscope (with atomic resolution, 0.8 Ångström) and **(ii)** a focused ion beam sample preparation system with scanning electron microscope monitorization (Tescan). A complex SPM scanning microscopy system **(iii)** consisting of AFM, MFM, STM, CFM, EFM, and nanoindentation, functioning under vacuum and at variable temperature (NT-MDT) was acquired as well.

A2. The Cleanroom
This infrastructure consists of **(i)** a class ISO 1000 cleanroom with an area of 45 m^2, where **(ii)** a Raith-Hitachi SEM nanolithography system with laser interferometry sample alignment, chemical huts and **(iii-iv)** two metallization systems with multiple cells for direct and electron beam evaporation, one for noncontaminated and one for contaminated metals, have been installed. Another cleanroom, of class ISO 100 and with an area of $15m^2$, houses **(v)** a new photolithography system (EV Group).

2.2. The Centre also planned the modernization of five existing laboratories:

B1. The Surface and Interface Science Laboratory, through the acquisition of **(i)** an angle- and spin-resolved photoelectron spectroscopy system and **(ii)** a LEEM-PEEM low energy electron and photoelectron microscopy system, both produced by Specs. The resulting complex system for the study of surfaces and interfaces is among the most performant existing in the European Union; regarding the LEEM-PEEM system, up to this moment there are only two such systems in Europe and five in the United States.

B2. The Complex Structural Materials Characterization Laboratory was modernized through the acquisition of (i) a pulsed Fourier Transform Electronic Paramagnetic Resonance (EPR) Spectrometer, which is unique at national level, (ii) a magnetic field Mössbauer spectrometer with a liquid helium cryostat (also unique) and (iii) a X-ray absorption spectrometer that makes it possible to accomplish at NIMP measurements that otherwise would require access to synchrotron radiation facilities (there is only one other system installed in Europe at this moment).

B3. The High Frequency (THz) Studies Laboratory, for which (i) a vector network analyzer from Agilent, an anechoic chamber and (ii) a spectrometer that can operate at up to 7THz (Aispec Japonia) were acquired. The latter acquisition is unique in the world; there has been no other such spectrometer sold in the United States, the European Union or any other developed economy with the exception of Japan. A variable temperature measurement station for small dimension samples (ii) was also acquired.

B4. The Optics and Spectroscopy Laboratory was modernized through the acquisition of (i) a Raman spectrophotometer with optical microscope operating in a large range of excitation energies, which constitutes, together with the existing equipment, the most important Raman measurement platform in Eastern Europe. A near field fluorescence optical microscope (ii) that can operate at liquid He temperatures, which is also a unique system, was acquired as well.

B5. The Complex Electrical and Magnetic Characterization Laboratory was completed with the following equipment: (i) a ferroelectric thin film tester; (ii) a cryoprober with micromanipulators; (iii) a cryogenic PPMS system for physical property measurements; (iv) a SQUID superconducting quantum interference magnetometer (minimum temperature of 2 K); (v) two helium liquefaction stations, one for each wing of the NIMP building.

3. DESCRIPTION OF THE MAIN EQUIPMENTS

3.1. Atomic resolution analytical electron microscope and dual system with scanning electron microscope and focused ion beam processing unit (SEM-FIB)

The technical performance of this complex equipment is at the highest level in the world up to date, being unique in Eastern Europe at this moment. The JEM ARM 200F electron microscope (Fig. 1) is operated at a maximal accelerating voltage of 200kV and is provided in standard configuration with a field emission gun (FEG) and a corrector of the spherical aberration (Cs corrector). The presence of the Cs corrector allows getting a space resolution of 0.08nm resolution in the STEM (Scanning Transmission Electron Microscopy) working mode.

The instrument performance and complexity are completed by the latest generation EDS (Energy Dispersive X-ray Spectroscopy) and EELS (Electron Energy Loss Spectroscopy) analytical units, allowing compositional measurements of extremely high precision both from the quantitative and space resolution point of view, going down to atomic space resolution. The instrument may be operated in at least 10 different working modes, such as: TEM/HRTEM, selected area electron diffraction (SAED), convergent beam

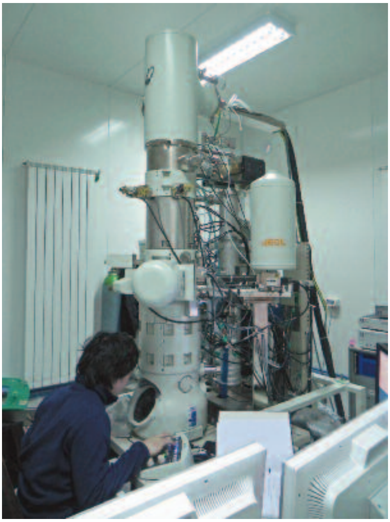

Fig. 1. JEM ARM 200F electron microscope (left); Dual system SEM-FIB Tescan Lyra III XMU (right)

electron diffraction (CBED), electron nanodiffraction, STEM, EDS in spot, line profile and 2D mapping modes (1 nm space resolution), EELS in spectroscopy or energy filtered imaging modes (spectrum image, EFTEM) at a space resolution down to atomic level. Equipment operation as well as data acquisition and processing are performed using complex dedicated software installed on the computers driving the microscope.

Beside the mentioned accessories fitting the microscope column (EDS, EELS, CCD cameras), a series of specialized devices and latest generation installations for TEM specimen preparation has been also acquired, the most complex of which being the SEM-FIB dual system Tescan Lyra III XMU. The SEM-FIB system is, in its turn, fitted with supplementary equipments allowing structural and compositional investigations, the EBSD (Electron Backscattered Diffraction) and EDS units, respectively.

3.2. Complex measurement system of magnetic, electronic and thermal properties of solids at low temperatures and in high magnetic fields

The complex system is composed of a QD-LHe-P18 liquid Helium plant – Cryomech, USA – and other two measuring systems (Fig. 2):

a) a magnetic properties measurement system, QD-MPMS-XL-7AC – Quantum Design (USA), which uses the state of art SQUID technology in order to achieve a high sensitivity (10-8emu) of the magnetic measurements in

Fig. 2. MPMS-SQUID component (left) and PPMS component (right)

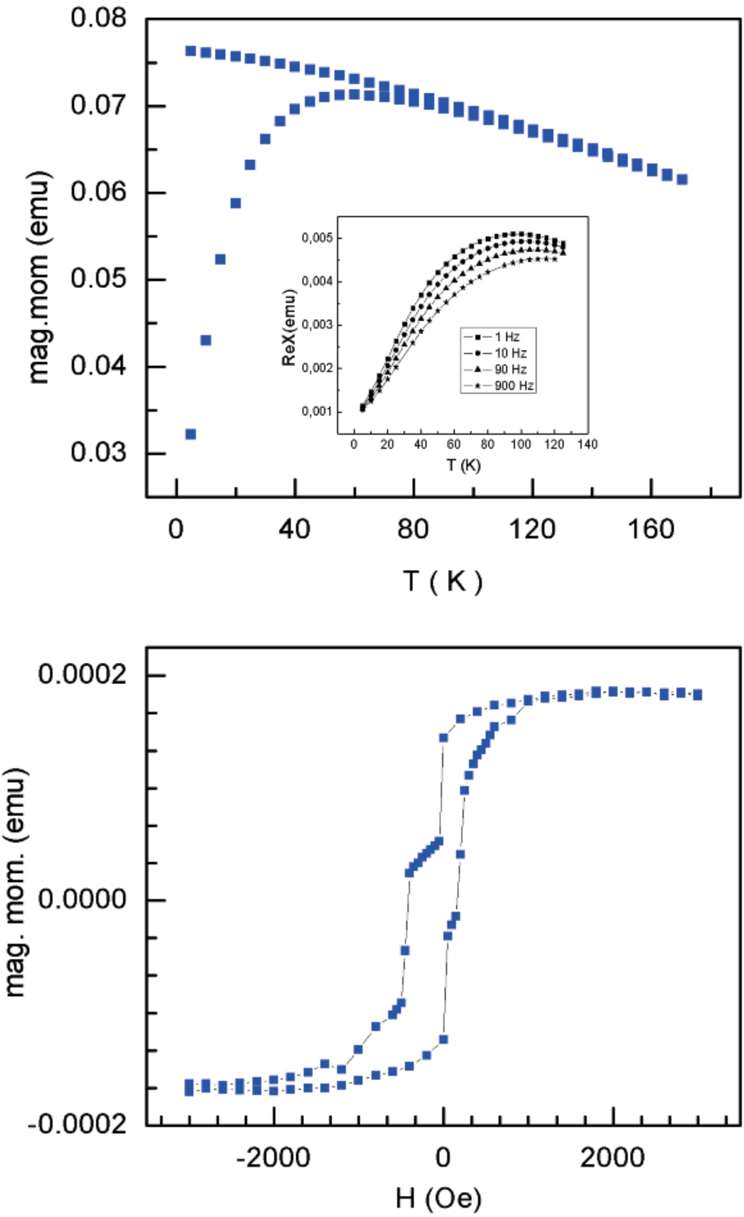

Fig. 3. ZFC-FC and AC susceptibility – in the insert – for a ferrofluid (left); hysteresis loop for a spin valve structure (right)

applied fields of up to 7T and over a temperature range from 2ºK to 300ºK and

(b) a physical properties measurement system QD-PPMS-14- Quantum Design (USA), optimized in order to combine magnetometry measurements with both thermal and electron-transport properties in applied fields of up to 14T and over a temperature range from 2ºK to 1000ºK.

The new characterization possibilities provided by this system will open additional opportunities for studying new materials and phenomena, like as antiferromagnetism and interfacial coupling in low dimensional systems, nanomaterials and multilayers, superconductors, magneto-optic and magnetoresitive structures, molecular coumponds, organic materials and composites with applications in electronics, biophysics, magnetochemistry and biology. Some of the already obtained results are presented in Fig. 3.

3.3. Experimental Cluster for Surface and Interface Science

The Cluster represents one of the most complex such systems in Europe, makes possible the preparation and characterization *in situ* of surfaces and interfaces, and consists of four units, of which the first three are mutually coupled (Fig. 4):

- The MBE (Molecular Beam Epitaxy) Chamber;
- The STM (Scanning Tunneling Microscope) Chamber;
- The Spin- and Angle-resolved Photoelectron Spectroscopy (SARPES);
- The PEEM (Photoemission Electron Microscopy) and LEEM (Low Energy Electron Microscopy) System.

All devices operate in ultra-high vacuum (1-2 x 10-10 mbar). It is possible to perform in situ characterization by low energy electron diffraction (LEED), reflection high energy electron diffraction (RHEED), Auger electron spectroscopy (AES), quadrupole mass spectroscopy (QMS), classical and high-resolution X-ray photoelectron spectroscopy (XPS), ultraviolet photoelectron spectroscopy (UPS), etc.

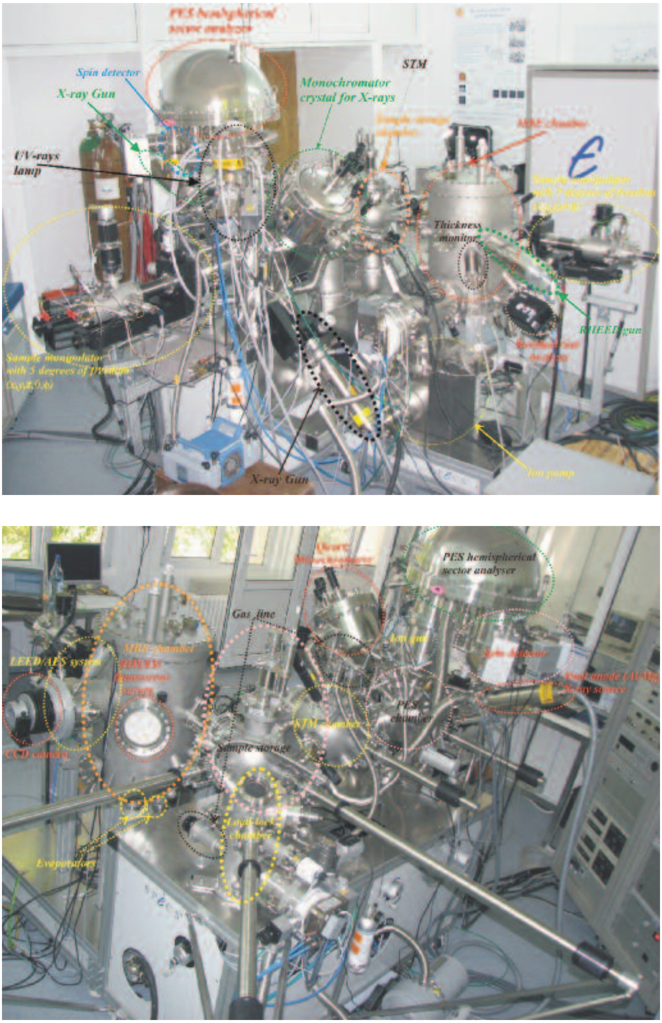

Fig. 4. Experimental measurement cluster for surface and interface science

The performance of the complex system described above is illustrated in Fig. 5 for high resolution photoelectron spectroscopy and in Fig. 6 for scanning tunneling miscroscopy with atomic resolution. Further, Fig. 7 shows the first results obtained for the visualization of the monatomic terraces of Si(001) by low energy electron microscopy (LEEM).

In approximately one year and 3 months of operation of the surface and interface facility, there has been a significant number of scientific papers published in ISI ranked journals with significant impact factor:

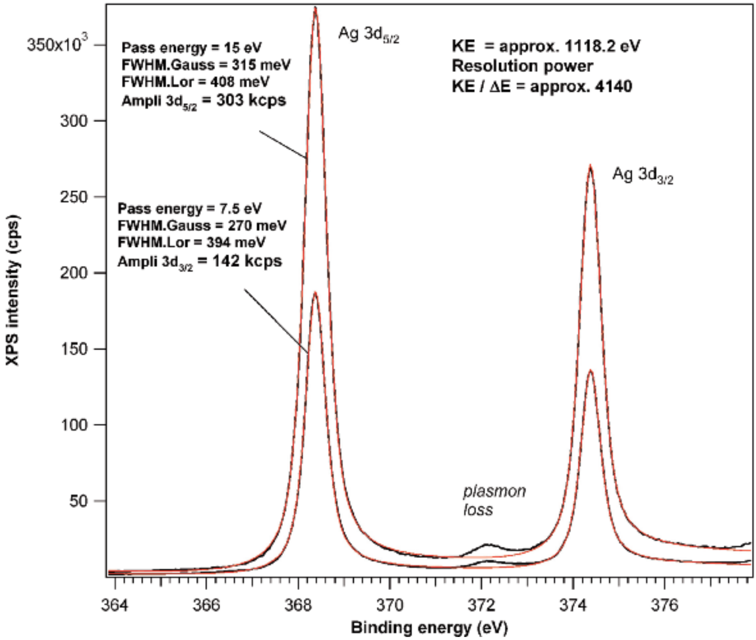

Fig. 5. High resolution XPS spectra for an Ag foil using a monochromatized X-ray source

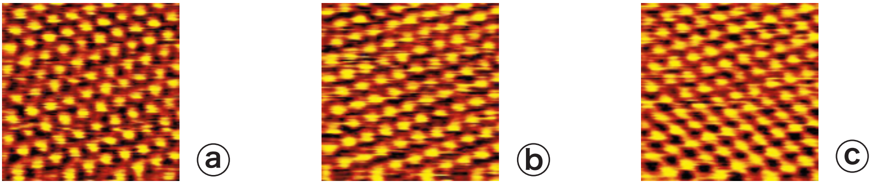

Fig. 6. Scanning Tunneling Microscopy (STM) images with atomic resolution obtained on highly oriented pyrolytic graphite (HOPG) (0001), with different tip voltages: **(a)** 630 mV; **(b)** 713 mV; **(c)** 884 mV

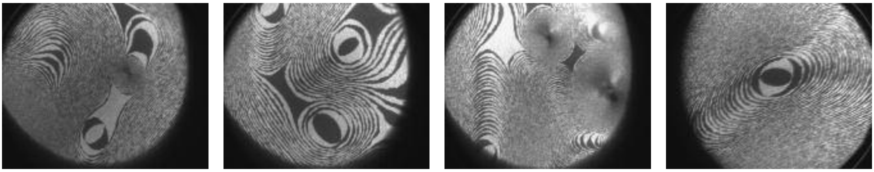

Fig. 7. LEEM darkfield images on one of the diffraction spots (1/2, 0) of the Si(001) surface reconstruction. The size of the image (field of view, FOV) is 3 μm

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Furthermore, these systems have made it possible to initiate the CNCSIS-PCCE Project *Surface and Interface Science: Physics, Chemistry, Biology, Applications (ID_76)* in satisfactory conditions as well as win two IFA-CEA cooperation contracts in 2010.

3.4. Pulsed Fourier Transform X-band ESR spectrometer

This spectrometer (Fig. 8) is a high performance instrument for cw (continuous wave) and pulsed ESR measurements in the 3.8 - 300 K temperature range. It is also equipped with pulsed ENDOR and ELDOR units for multiresonance experiments. This instrument is dedicated to the investigation of paramagnetic atomic and molecular species in various materials: crystalline and amorphous semiconductors, insulators, superhard materials, glasses, biomolecules, chemically active molecular species etc. It completes the multifrequency cw ESR equipment of the Center for advanced ESR techniques (CetRESav) from NIMP (www.cetresav.infim.ro) in performing multifrequency and multiresonance ESR investigations on a broad range of materials and nanomaterials.

The main operating parameters are: **(i)** microwave frequency range (cw mode): 9.2–9.9GHz; **(ii)** central frequency (pulse mode): 9.7 GHz; **(iii)** RF

Fig. 8. Pulsed Fourier Transform X-band electron spin resonance (ESR) spectrometer model Bruker BioSpin ELEXSYS 580 10/12

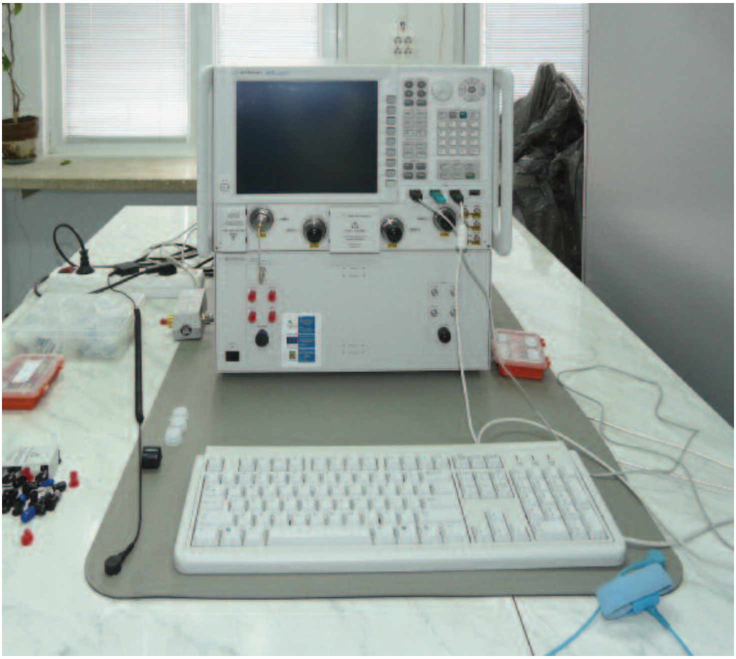
frequency range and power (for ENDOR measurements): 100kHz – 250MHz, 150 W; **(iv)** magnetic field range: 0,03–1,45T; **(v)** sensitivity (cw mode): 1.2x109spins/Gauss; **(vi)** pulse resolution: 1nsec; **(vii)** microwave peak power (pulse mode): max. 1kW.

3.5. Vector Network Analyzer and Terrahertz Time-Domain Spectrometer

The main piece of the equipment is the Agilent PNA-X N5245A Vector Network Analyzer shown in Fig. 9, which is equipped with 4-ports, dual source, internal combiner and mechanical switches, extended power range and bias-tees, frequency offset and added IF inputs for antenna and millimeter wave characterization. The system allows several types of measurements: **(i)** S parameters (magnitude and phase) in frequency domain; **(ii)** nonlinear component characterization, **(iii)** nonlinear X-parameter, **(iv)** time domain measurements.

As standalone, the PNA-X operates in the 10MHz to 50GHz frequency range. However, the N5261A 2-Port Millimeter-wave Test Set Controller and

Fig. 9. Agilent N5245A PNA-X Vector Network Analyzer



the millimeter-wave heads extend the measurement frequency range towards much higher frequencies, up to 500 GHz. The accuracy of the measured data is assured by the adequate calibration with a coaxial electronic calibration module (10MHz to 67GHz) and waveguide calibration kit for each frequency band of the millimeter-wave heads.

The nonlinear measurement capabilities of the system allow characterization of nonlinear materials (such as ferroelectrics and multiferroics) and nonlinear devices.

In the same context, a TerraHertz time domain spectrometer (Aispec pulse IRS2000 pro) (Fig. 10) was acquisitioned in order to carry out measurements in a very wide frequency band from 40GHz up to 7THz. The spectrometer allows several types of measurements: **(i)** transmission measurements in the –180°C to +300°C temperature range, **(ii)** reflection measurements, **(iii)** Attenuated Total Reflection (ATR) measurements , measurements in liquid and gas samples.

The measurements are controlled by the computer using the "Aispec THZEQ Measurement Program". The software set the instrument for the wanted measurement type and carries out the data acquisition. The time domain data are transformed in frequency domain by using Aispec IR

Fig. 10. TerraHertz time domain spectrometer (Aispec pulse IRS2000 pro)

Spectra – Infrared Spectroscopy Data Analysis Program. This software provides transmittance or reflectance data, as well as material data as the complex refractive index versus frequency, dielectric constant and losses, conductivity etc.

3.6. Cleanroom for preparation and characterization of nanostructured materials samples

Within the frame of CEUREMAVSU project, a cleanroom facility (Fig. 11), has been established in NIMP. The cleanroom, necessary for preparation and characterization of samples of nanostructured materials, is fitted with the necessary infrastructure for top performance.

The cleanroom is designed, built and exploited according to the international standards specific to the field, especially ISO EN 14644, concerning the classification of such a construction after the characteristic parameters as temperature, humidity and number of small particles in the unit of volume.

The cleanroom facility is composed by two sections: one section class 100 for photolithography and another one, class 1000, dedicated to

Fig. 11. Cleanroom at NIMP for preparation and characterization of nanostructured materials and nanostructures

Fig. 12. Nanolithography facility fitted to a Scanning Electron Microscope (SEM) set in to function in the class 1000 section of the NIMP cleanroom

technological operations, such as wet etching and metallization and also for sample prepation in equipments such as nanolitography assisted by SEM (Scanning Electron Microscope) and by focused ion beam (FIB). These equipments allow also preliminary characterization of sample surface.

The EVG620 mask aligner is a complex equipment for photolithography which meets the demanding requirements of advanced research in the field of nanostructured materials. This is a state of the art equipment with sub-micron resolution for photolithography, using an Ultra-Violet (UV) light source and is allows also Deep Ultra-Violet (DUV) operation for better resolution. As a plus, this equipment is fitted also with a NanoImprint Lithography (NIL) facility, another modern technological operation in preparation of nanostructures. This operation allows synthesis on a substrate of a two-dimensional structure with sub-micron resolution, using a pre-defined „stamp".

The SEM equipment with nanolithography facility (Fig. 12) brings together the advantages of the top quality SEM Hitachi S-3400N with a one of a kind Elphy Quantum nanolithography facility, produced by the world leader, Raith company.

This system is fitted also with a "beam blanker" module and a LST-X laser interferometry positioning stage, both produced by Deben UK Limited. The resolution of a SE image is minimum 3.0 nm at 30 kV accelerating voltage (x 100,000, WD = 5mm, High Vacuum mode) and the resolution of BSE image is minimum 4.0nm at 30kV (x 60,000, WD = 5mm, Low Vacuum mode). The magnification may be adjusted continuously in the range of x 5 to x 300,000.

The deposition of thin films, metal or oxides, is a mandatory operation for synthesis on samples of electrical contacts or insulating areas for complex characterization of functional structures of nanostructured materials, characterization performed by the means of measuring the electrical, electro-optical and galvanomagnetic parameters. For this operation, a deposition system was purchased and fitted in the cleanroom. This equipment is comprised of two modules: one modul for deposition of usual CMOS-compatible materials, and the second module for deposition of materials with contamintion potential.

4. CONCLUSION

All these equipments, added to the existing new ones (the estimated total investment in NIMP in the period 2004–present being around € 18 Million), together with the acknowledged international level of NIMP's researchers, promote the Institute at a top level in the area of European Research in Materials Physics and Condensed Matter Science. NIMP therefore confirms its top ranking in Romanian Physics Research, as it was constantly demonstrated since several decades by the impact factor of NIMP publications, normalized to the number of researchers.

One may stress that already at present, when the benefits of the POS-CCE just started, the June 2010 Ranking Web of World Research Centres (a CSIC-Spain initiative, see http://research.webometrics.info), when dealing with research centres, places NIMP (ranked 1120) in the second Romanian position, after the National Institute of Nuclear Engineering IFIN HH (ranked 946) and even before the impressive Mathematics Institute of the Romanian Academy (ranked 1585) and before the Romanian Academy (ranked 2078). Therefore, NIMP is present in the Top 4000 of this ranking.

Also, in another recently published ranking (SCImago Institutions Rankings 2009 World Report” published on http://www.scimagoir.com), NIMP is ranked 1585 (the second position at National Institutes), respectively the fifth position at the national level, by taking into account the general output (including any kind of publications).

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