IAP Accelerator Based Microanalytical Facility for Material Science Studies

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Abstract. Microanalytical facility developed at the Institute of Applied Physics, National Academy of Sciences of Ukraine (IAP) based on a single-ended 2 MV Van de Graaf accelerator. It consists of 4 beamlines to fulfill materials science tasks using following techniques: Rutherford backscattering, nuclear reaction analysis, ion luminescence and particle induced X-ray emission. Rutherford backscattering beamline is equipped with magnetic spectrometer with double focusing and resolution of 3 keV at Ep=1 MeV. A scanning nuclear microprobe is implemented in one of the beamline. Proton beam focusing is realized with two doublets of magnetic quadrupole lenses of a new design. Both doublets are made of single piece of the soft iron using electrical-discharge machining system. Resolution of 2 μm with current about 100 pA is obtained. The microprobe is used for investigation of the impurity segregation to the grain boundary in the structural materials of the nuclear power systems. Article presents a description of the facility and its applications.

1. Introduction

Institute of Applied Physics, National Academy of Sciences of Ukraine (IAP) realizes project of the nanoanalytical center creation using electrostatic accelerators. The main purpose of this center is to conduct research into the structure and composition of the reactor materials with high depth and surface resolution of the sample as well as investigation of the defect material structure subjected to irradiation. Core of the center is the following facilities: microanalytical complex based on the 2MeV electrostatic accelerator designed and constructed at IAP, AMS-4130 accelerator mass spectrometer manufactured by HVEE, Netherland, 6 MV Pelletron analytical facility transferred by the Max-Planck Institute, Stuttgart, Germany, laser mass spectrometers and laser emission spectrometer with inductive coupled plasma developed at IAP.
This report is focused mainly on the microanalytical facility based on a single-ended 2MV Van der Graaf accelerator developed and commissioned at IAP in 2008.

2. Electrostatic accelerator

Fig. 1 shows general view of the electrostatic accelerator designed and constructed at the National Research Center "Kharkov Institute of Physics and Technology", National Academy of Sciences of Ukraine specially for the analytical application in nuclear science and technology [1,2]. The first version of this facility included the analyzing magnet with bending function in order to decrease its cost. Vacuum system and beam transport system were maximum simplified. This limits analytical capacities and application field of the facility. During the last few years modernization and further development of the facility, i.e. entirely replacement of the vacuum system, improvement of the beam transport system and construction of new analytical channels have been accomplished at IAP. In the framework of updating analyzing and bending magnets and quadrupole electrostatic lenses were designed at IAP and manufactured by Research Institute of

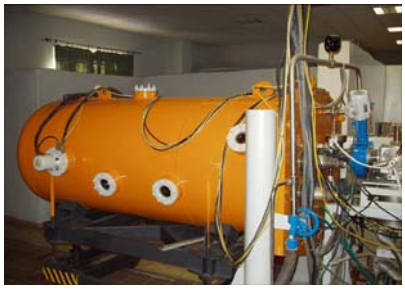

FIG. 1. 2 MV electrostatic accelerator.

Electrophysical Equipment, St.Petersburg, Russian Federation. Electrostatic accelerator is intended for the proton beam and helium ion production and characterized by the following performance data:

- energy, MeV 0.3-2.0
- energy stability, % 0.1
- ion type H^+, He^+
- proton beam current, μA up to 50
- operating mode continuous
- weight, tonne 3.0

In order to obtain high quality ion beams accelerator construction was modified by using ion source with permanent magnet system to increase plasma density and preliminary beam focusing [3,4], and by reconstruction of the high-voltage column and charge transporter improvements [5,6]. Fig. 2 shows block scheme of the microanalytical facility of IAP.

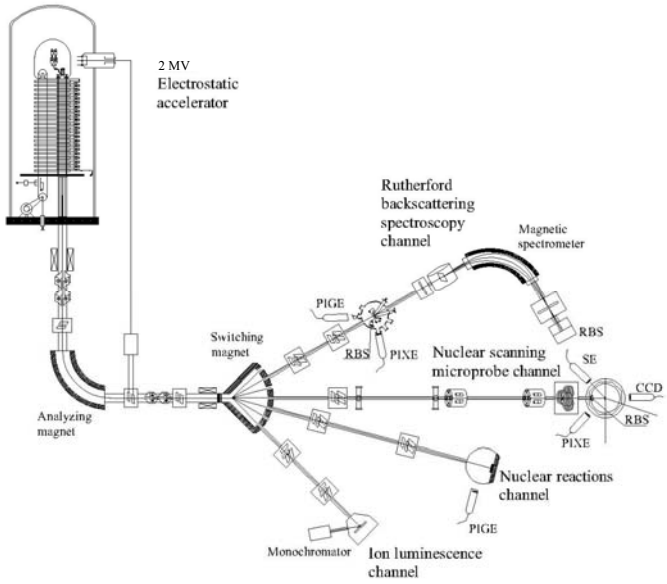

FIG. 2. Block scheme of the microanalytical facility of IAP.

3. Analytical end stations

Fig. 3 shows general view of the analytical channels of the microanalytical facility. The facility includes four analytical channels, i.e. nuclear scanning microprobe channel, Rutherford backscattering channel, nuclear reaction channel and ion luminescence channel.

FIG. 3. Analytical channels of the microanalytical facility.

Rutherford backscattering channel with high resolution

Magnetic spectrometer with double focusing described in [7] was taken as the base of the facility. General view of the spectrometer is shown in Fig. 4.

FIG. 4. Spectrometer.

This spectrometer with sector-shaped unified magnetic field has the following parameters: radius of the particle trajectory curvature in magnetic field is 320 mm, shear angle at the entrance and outlet are 46^0 and $4^051'$, respectively, gap width is 16 mm, gap height is 106 mm, distance from the source (target) to entrance into the magnet and from the magnet outlet to the detector are 400 and 700mm, respectively. Measured value of the spectrometer solid angle is $3.56\pm0.23\cdot10^{-3}$ steradian. Magnet with the scattering chamber are mounted on the platform permitting to rotate it $0\text{-}150^0$ to the left and $0\text{-}20^0$ to the right related to the incident beam.

The facility was equipped with precision power supply [8], microprocessor controller of the data acquisition, magnetic field measurer, current integrator, power supply of the charged particle detector and preamplifier for this detector. All listed units of the spectrometer except

of the magnet, platform and some parts of the vacuum system were designed and constructed at IAP. The vacuum pumping system of the spectrometer was also modified. In addition, software permitting to automate control and data acquisition during the experiment was developed.

Fig. 4 presents dependence of the device energy resolution on the slit width measured on a silicon sample with proton energy of 1MeV.

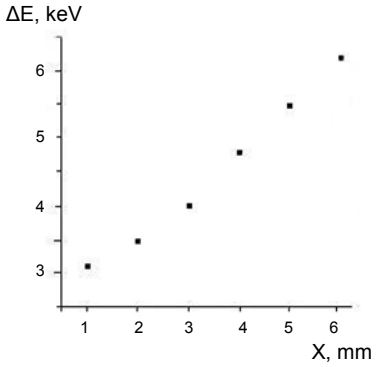

FIG. 4. Energy resolution vs slit width.

Resonance nuclear reaction channel

The channel is intended for the impurity distribution profile depth measurements in the materials. Using of the proton resonance reaction allows to obtain high spatial resolution. Table 1 presents data on the detection and spatial resolution limits of this technique. This channel can be applied only for light elements.

TABLE I: DETECTION LIMITS AND LATERAL RESOLUTION FOR SOME RESONANT NUCLEAR REACTIONS

Element	Nuclear reaction	Resonance energy (keV)	Resonance width (eV)	Resolution (nm)	Detection limits ($1:10^6$)
Hydrogen	$^{1}H(^{15}N,\alpha\gamma)^{15}C$	6385	6000	4	30
	$^{1}H(^{19}F,\alpha\gamma)^{16}O$	6421	45000	23	100
Lithium	$^{7}Li(p,\gamma)^{8}Be$	441	12200	220	0,15
Boron	$^{10}B(\alpha,p)^{13}C$	1507	18000	30	5000
	$^{11}B(p,\gamma)^{12}C$	163	5200	130	50
	$^{11}B(p,\alpha)^{8}Be$	660	300000	50	100
Carbon	$^{12}C(p,\gamma)^{13}N$	457	31700	650	
	$^{13}C(p,\gamma)^{14}N$	1748	135	330	
Nitrogen	$^{14}N(\alpha,\gamma)^{18}F$	1531	600	2	
Oxygen	$^{18}O(p,\gamma)^{15}N$	898	2200	5	5
		633	2100	20	
Fluorine	$^{19}F(p,\gamma)^{16}O$	340	2300	25	0,1
Natrium	$^{23}Na(p,\gamma)^{20}Ne$	592	600	15	0,5
Magnesium	$^{24}Mg(p,\gamma)^{25}Al$	223	<32	0,6	
Aluminium	$^{27}Al(p,\gamma)^{28}Si$	405	87	1,1	100
		992	105	10	
Phosphorus	$^{31}P(p,\gamma)^{28}Si$	1018	<300	10	100
	$^{31}P(p,\gamma)^{32}S$	1147	<160		50
Argon	$^{40}Ar(p,\gamma)^{41}K$	1102	90	7	2000
Titanium	$^{48}Ti(p,\gamma)^{49}V$	1361	50	10	20
Chrome	$^{52}Cr(p,\gamma)^{53}Mn$	1005		9	100

Ion luminescence channel

The channel includes a crystal monochromator operating in the wavelength range of 300…800nm. The channel is applied to investigate chemical composition of impurities in materials.

Nuclear microprobe facility

Microprobe channel [9] is positioned at a zero angle of the bending magnet (schematic arrangement is given in Fig. 5). All channel components are placed on two granite blocks (see position 1, Fig. 5).
Each block is of 500 kg weight and supported be concrete supports (2) placed on a sand cushion (3). The vacuum is maintained at 10^{-6} Torr by means of two turbo-molecular (4) and two ion pumps (5). During the measurements the vacuum is maintained by ion pumps only to avoid vibrations.

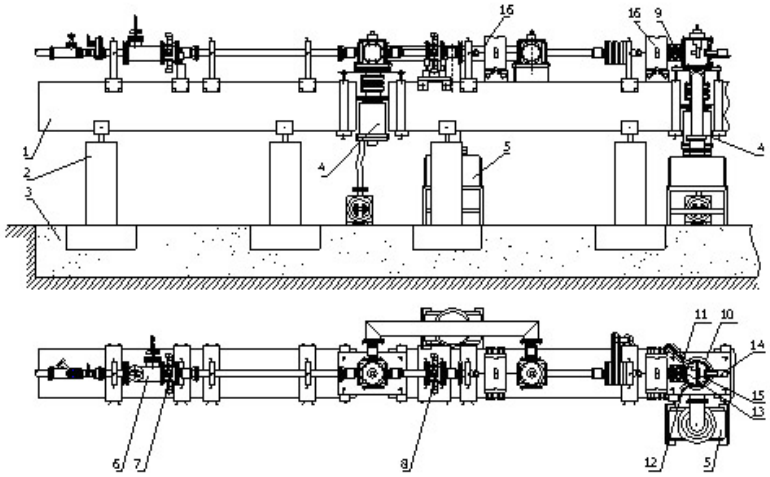

FIG. 5. Scheme of the nuclear microprobe units and systems.

The beam collimation is performed using a preliminary (6), object (7) and angular (8) collimators. The preliminary collimator of slit-type with tantalum slit edge and water cooling is intended to decrease the beam current at the entrance of the object collimator. The object and angular collimators are two mutually perpendicular slits of similar design. Collimating slit edge is a circular polished rod of 5mm diameter made from tungsten. Each slit edge is driven independently by a mechanical differential microscrew with a positioning accuracy of 2μm and can be moved within the range from 0 to 2mm.

FIG. 6. Doublet of magnetic quadrupole lenses of new design.

The beam focusing on the target is realized by the microprobe forming system based on a separated "Russian quadruplet" with two integrated doublets of magnetic quadrupole lenses of new design (16) [10]. Yoke and poles of each doublet are produced of a single peace of soft magnetic iron by electric erosion technique. This allows to provide quadrupole field symmetry and high precision of the lens adjustment.

The scanning system (9) consists of four ferromagnetic coils. The coil commutation (X, Y,Y, X) provides a conventional scanning mode of the focused beam in the range of ±500 μm for the post-lens arrangement of the scanning system with frequency up to 5kHz. Also (X,Y, -Y,-X) commutation permitting beam-rocking scanning mode is possible. To control beam current in the scanning coils a specific power supply was developed which was synchronized with the data acquisition system [12].

The chamber (10) is shaped like an octahedron. In the vertical chamber walls there are windows with unified flanges carrying a secondary electron detector (11), PIXE detector (semiconductor detector AMPTEK® (XR-100CR)), scanning system (9), circular charged particle detector (annular surface-barrier detector ORTEC® (TC-017-050)) (13) and CCD-equipped optical microscope (14). Target positioning system has two degree of freedom (X-Y) (15). Target holder can hold up to 16 samples.

Microprobe resolution in the microanalysis mode was determined using focused beam scanning of the standard copper net with step of 1000 cell per inch and further detection of the secondary electron emission. Fig. 7a shows secondary emission image of the single cell. Figs. 7b,c show secondary electron yield during the scanning in X and Y directions. Processing of the secondary electron yield diagrams shows that microprobe resolution in the microanalysis mode with I~150pA is 2 μm (FWHM).

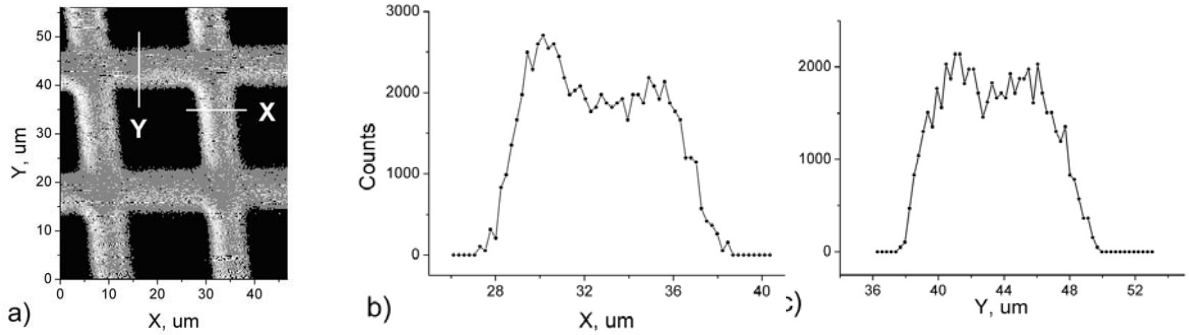

FIG. 7. a) single cell of the copper net with step of 1000 cell per inch; b), c) secondary electron yield in X and Y directions, respectively.

4. Future plan

Construction of the AMS isotope mass spectrometer, model 4130, manufactured by HVEE, Netherlands, based on the 1MV tandem accelerator is scheduled by IAP for this year. Equipment has been already supplied to the Institute and commissioning is planned to be start in May this year. The mass spectrometer is expected to be used for carbon-14 dating with following application in materials science, environmental protection, medicine etc. Fig. 8 displays mass spectrometer units inspection after their transportation to IAP.

FIG. 8. Mass spectrometer units inspection after their transportation to IAP.

FIG.10. Pelletron-6 accelerator-based system.

Radiative study of materials requires development of new experimental basis to investigate radiative defects of the reactor materials. Measurement of positron life time in materials is effective method for the low-sized defects examination. Max Planck Institute, Stuttgart, Germany has transferred to IAP an analytical facility based on the Pelletron-6 accelerator permitting to obtain positron beams with variable energy up to 6MeV. A general view of the accelerator before its demounting and transportation to IAP is presented in Fig. 9. This accelerator is equipped also with analytical channel to measure hydrogen and helium distribution profiles in the reactor materials. This area becomes topical due to development new materials for the fast neutron reactors and the ITER fusion reactor. Analytical parameters of this channel are presented in Table 2 [13].

TABLE II: ANALYTICAL PARAMETERS OF THE PELLETRON-6 ACELERATOR

Methods	Type	Ions	Application	Sensitivity	Resolution
RBS	Standard	1-2 Mev He	med., heavy	10 atppm	10nm
	Special		med., heavy	0.1 atppm	50nm
	Special		med., heavy	1000 atppm	0.1nm
ERDA	Standard	3 Mev He	H	10 atppm	100nm
	Special	4.5 Mev Ne	H	1000 atppm	0.5nm
	Special	1.5 Mev Ar	Light atoms	1000 atppm	0.2nm
NRA	Standard	p, d, 3He	Light atoms	10-1000 atppm	100nm
PIXE	Standard	3MeV p, He	No light atoms	1-10 atppm	none

5. Conclusion

Due to the necessity to shorten a long cycle of the new reactor material development novel fields of the MeV energy accelerators application appear. These accelerators are used for simulation of the radioactive defects detection, hydrogen and helium content in the reactor materials. This allows to reduce defects accumulation material test time significantly and to select the most perspective materials for intrareactor tests. The second field of the MeV accelerator application is validation of the modern potentials and different empirical parameters used while computer simulation of the material radioactive damages (ab-initio, molecular dynamics, Monte-Carlo etc.). Precision electrostatic accelerators and well-proven nuclear methods open possibilities for mentioned investigations and supplement possibilities of the synchrotron radiation sources and SNS facilities while their considerably smaller cost and affordability for the research institutes and universities. High resolution is of great importance for measurements of impurity distribution and damages 3D profiles in the materials. To date available resolution over surface is of 0.1μm and depth resolution is of nanometre values. Existing photon and neutron sources are far to reach these possibilities.

6. References

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