Zał. nr 5 do ZW 78/2023

Attachment no. 4. to the Program of Studies

FACULTY of Chemistry

SUBJECT CARD

Name of subject in Polish *Zaawansowane metody badawcze w inżynierii materiałów*
Name of subject in English Advanced research methods in the engineering of materials
Main field of study (if applicable): *Chemistry and engineering of materials*
Specialization (if applicable): Advanced Nano and Biomaterials - MONABIPHOT
Profile: *academic*
Level and form of studies: *2nd level, full-time*
Kind of subject: *obligatory*
Subject code W03ANB-SM2013W, W03ANB-SM2013C, W03ANB-SM2013L
Group of courses NO

	Lecture	Classes	Laboratory	Project	Seminar
Number of hours of organized classes in University (ZZU)	30	15	15		
Number of hours of total student workload (CNPS)	50	25	50		
Form of crediting (Examination / crediting with grade)	Exam	Crediting with grade	Crediting with grade		
For group of courses mark (X) final course					
Number of ECTS points	2	1	2		
including number of ECTS points for practical classes (P)		1	2		
including number of ECTS points corresponding to classes that require direct participation of lecturers and other academics (BU)	1,3	0,7	0,7		

*delete as not necessary

PREREQUISITES RELATING TO KNOWLEDGE, SKILLS AND OTHER COMPETENCES

1. Basic knowledge about the structure of the atom and chemical bonds.
2. Basic knowledge about the structure of popular engineering materials.
3. Basics of electrochemistry, the concept of electrochemical potential, the phenomenon of electrochemical corrosion.
4. Basics of the interaction of solids with different types of radiation. Principles of spectroscopic techniques.

SUBJECT OBJECTIVES

C1. Understanding the importance of solid surface in nanotechnology.
C2. To acquaint students with modern and advanced techniques of surface research, morphology and structure of engineering materials.
C3. Ability to choose an appropriate method of determining: surface composition, surface topography, adhesion and hardness to the material being tested.

C4. Understanding the interaction of the surface of the material with the corrosive environment.
C5. Ability to apply standards in making measurements and their statistical treatment.

SUBJECT EDUCATIONAL EFFECTS

related to knowledge:

PEU_W01 Student has elementary theoretical knowledge about XPS / AES methods and equipment working in ultra high vacuum.

PEU_W02 The student has a basic knowledge of the research possibilities of determining the surface chemical composition by XPS and AES.

PEU_W03 Student ma podstawową wiedzę o mikroskopii elektronowej (SEM) oraz mikroanalizie rentgenowskiej (EDS), a także o systemie orientacji i detekcji faz na podstawie dyfrakcji elektronów wstecznie rozproszonych (EBSD).

PEU_W04 The student has a basic knowledge of the method of determining the mechanical properties of materials based on the measurements of microhardness and adhesion, as well as on the method of determining geometric parameters of the surface.

PEU_W05 The student has a basic knowledge of the types of electrochemical corrosion and laboratory techniques for testing the corrosion resistance of materials. The student has a basic knowledge of electrochemical impedance spectroscopy (EIS).

PEU_W06 The student knows the basics of impedance spectroscopy to determine the material properties of dielectric materials.

PEU_W07 The student knows the basics of structural analysis by X-ray diffraction (XRD) in the analysis of metals, their alloys and ceramics.

related to skills:

PEU_U01 Student is able to characterize qualitatively and quantitatively the tested surface of a solid material.

PEU_U02 The student is able to perform basic operations on XPS spectra and use the XPS and AES databases for the purpose of qualitative interpretation of XPS, AES spectra.

PEU_U03 The student is able to select the parameters of the scanning microscope (SEM), microanalysis (EDS) and the EDS and EBSD databases that are appropriate for the material being studied.

PEU_U04 The student is able to perform a DC polarization measurement and is able to determine the basic electrical quantities that characterize the corrosion process.

PEU_U05 The student can perform the measurement using the EIS technique, analyze and interpret the simplest impedance spectrum, propose an electric substitute circuit.

PEU_U06 Student is able to determine the basic properties of dielectric material using impedance spectroscopy.

PEU_U07 The student can interpret the XRD diffractogram.

PEU_U08 Student based on available standards can interpret the type of damage of the coating during scratch-test adhesion measurements.

PEU_U09 The student is able to register the surface profile of the tested material and determine on its basis the most important geometric parameters of the tested surface.

PEU_U10 The student is able to measure the thickness of the coating / thin layer and interpret the dependence of the penetration depth on the applied force during microhardness measurement.

related to social competences:

PEU_K01 The student is ready to apply the acquired knowledge to solve research problems.

PEU_K02 The student understands the need to use expert knowledge when interpreting the obtained research results.

PROGRAMME CONTENT

	Lecture	Number of hours
Lec 1	XPS, AES - basic concepts: surface, spectroscopic and X-ray notation. Photoelectric process - primary and secondary emission.	2
Lec 2	X-ray photoelectron spectroscopy (XPS / ESCA). “Depth” in XPS / AES analyzes. Spectrum and its components. Stages of the analytical process. Auger electron spectroscopy (AES). Basic instrumentation (UHV, energy analyzer, photon source, ion gun, manipulator).	2
Lec 3	Practical applications of electron spectroscopy in materials engineering. Examples of spectra (XPS / AES) and their interpretation: in microelectronics, ceramics, catalysis, semiconductor and polymer materials, metallurgy, and corrosion of materials.	2
Lec 4	Basic polarization techniques for testing the corrosion resistance of materials. Measuring systems. Interpretation of current-voltage characteristics.	2
Lec 5	Electrochemical impedance spectroscopy (EIS). Impedance and methods of its presentation, basic concepts, available measurement techniques, spectra and their analysis, electrical equivalent models in the study of corrosion processes.	2
Lec 6	The profilometric methods for determining the surface topography of coatings and thin films.	2
Lec7	Determining the thickness of coatings and thin layers. Available non-destructive measurement techniques.	2
Lec8	Determination of microhardness of coatings and layers.	2
Lec9	Determination of adhesion of coatings and layers.	2
Lec10	Impedance spectroscopy of dielectric materials.	2
Lec11	Basics of electron microscopy (SEM) and X-ray microanalysis (EDS). Basic instrumentation (electron and X-ray detectors, vacuum system, preparations used in electron microscopy).	2
Lec12	Application of SEM, EDS and EBSD in material engineering. Examples of SEM images, spectra and chemical composition of EDS and crystallographic orientation maps (EBSD). Basics of spectra interpretation.	2
Lec13	Application of SEM / PFIB and TEM techniques in the analysis of the structure of coatings and thin films. Sample preparation. Contemporary analytical capabilities.	2

Lec14	Structural investigations of metals and their alloys as well as ceramic materials by X-ray diffraction (XRD).	2
Lec15	The use of GC-MS in determination of organic compounds.	2
	Total hours	30

Classes		**Number of hours**
Cl 1	Organizational matters.	1
Cl 2	Data treatment and analysis of linear polarization resistance characteristics. Determination of electrochemical parameters from polarization curves.	2
Cl 3	Interpretation of impedance spectra from EIS measurements. Proposing of physical models and electric equivalent circuits. Calculation of the values of elements of the electric equivalent circuit by non-linear least squares method.	2
Cl 4	Familiarization with software for interpreting XPS and AES spectra on the basis of real experimental spectra. Electron spectra: qualitative and quantitative interpretations. Acquisition of low- and high-resolution spectra.	2
Cl 5	Identification of spectral components. Quantitative calculations of elemental surface composition. Elimination of spectral by-products, methods for determining the background line. Calibration of spectra.	2
Cl 6	Calculating the average mean free electron path (IMFP) based on available models. Calculating the thickness of passive / oxide layers based on selected models.	2
Cl 7	Determination of dielectric properties of ceramics by dielectric impedance spectroscopy. Spectra processing and interpretation.	2
Cl 8	GC-MS technique. Analysis of chromatograms for organic compounds.	2
	Total hours	15

Laboratory		**Number of hours**
Lab 1	Organizational matters. Health and safety training.	1
Lab 2	Determination of the corrosion rate using linear polarization resistance and polarization curves.	2
Lab 3	EIS spectroscopic measurements of the corrosion process of selected metals and alloys.	2
Lab 4	Measurement of surface roughness by contact profilometry method. Profile registration and determination of basic geometric parameters of the surface based on the standard.	2
Lab 5	Measurements of coating thickness by means of magnetic induction and Eddy current methods.	2
Lab 6	Measurements of microhardness of coatings and thin films. Oliver and Pharr method. Statistical analysis of results. Testing of adhesion and resistance to scratch of metal coatings using the scratch-test method. Microscopic evaluation of the scratch track based on the standard.	2
Lab 7	Analysis of the surface morphology of the material by means of scanning electron microscopy (SEM). Secondary electron and back scattered electron imaging.	2

Lab 8	Quantitative analysis based on X-ray microanalysis (EDS) as well as map analysis of crystallographic orientation (EBSD) of ceramics, metals and semiconductors.	2
	Total hours	15

TEACHING TOOLS USED

N1. Multimedia presentation - thematic lecture.
N2. Practical exercises in the laboratory.
N3. Presentation / demonstration.
N4. Computer.
N5. Use of available scientific literature (Web of Science, Scopus), NIST database.
N6. The use of SpecLab, XPSPeak, Quases, Gamry, Nova, SAI, CSM, Bruker, FEI, TEAM, Origin software.

EVALUATION OF SUBJECT LEARNING OUTCOMES ACHIEVEMENT

Evaluation (F – forming during semester), P – concluding (at semester end)	Learning outcomes code	Way of evaluating learning outcomes achievement
F1 = P1 (classes)	PEU_W01- PEU_W07, PEU_U01- PEU_U10, PEU_K01-K02	Grade from the tests
F1 (laboratory)	PEU_W01- PEU_W07, PEU_U01- PEU_U10, PEU_K01-K02	Grade from the reports
F2 (laboratory)	PEU_W01- PEU_W07, PEU_U01- PEU_U10, PEU_K01 -K02	Grade from the tests
P (laboratory) Arithmetic average of F1 and F2 forming grades		
P (lecture)	PEU_W01- PEU_W07	Final exam

PRIMARY AND SECONDARY LITERATURE

<u>PRIMARY LITERATURE:</u>

[1] XPSPeak41 Manual.
[2] An Introducing to Surface Analysis by XPS and AES; J.F. Watts, J. Wolstenholme, John Wiley&Sons Ltd., 2003.
[3] Electrochemical Impedance Spectroscopy; Mark E. Orazem, Bernard Tribollet, John Wiley & Sons Ltd., 2011.
[4] Scanning Electron Microscopy and X-Ray Microanalysis 4th ed., Goldstein, J.I., Newbury, D.E., Michael, J.R., Ritchie, N.W.M., Scott, J.H.J., Joy, D.C., 2018.
[5] K. Nitsch, Zastosowanie spektroskopii impedancyjnej w badaniach materiałów elektronicznych, Oficyna Wydawnicza PWr, 1999.
[6] B. D. Cullity and S. R. Stock, Elements of X-ray Diffraction, Pearson, 2001.

<u>SECONDARY LITERATURE:</u>

[1] http://www.casaxps.com/ebooks/ebooks.htm

[2] Oliver W.C., Pharr G.M. *„An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments”*. Journal of Materials Research. Vol. 7, No. 6 (1992): pp. 1564÷1583.
[3] https://www.gamry.com/application-notes/EIS/basics-of-electrochemical-impedance-spectroscopy/
[4] https://www.bruker.com/products/surface-and-dimensional-analysis/stylus-profilometers/dektak-xt/learn-more.html
[5] https://blog.phenom-world.com/
[6] https://www.ameteksi.com/products/materials-testing-systems/1296a-dielectric-interface
[7] https://www.fei.com/products/sem/quanta-sem/

SUBJECT SUPERVISOR (NAME AND SURNAME, E-MAIL ADDRESS)

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