

# **COURSE CARD**

#### 1. Basic information

Course name in English:	Advanced research techniques in material engineering		
Course name in Polish:	Zaawansowane techniki badawcze w inżynierii materiałowej		
Number of hours:	30		
Type of course:	Elective course		
Form of course:	mixed forms (combination of lecture, seminar laboratory)	and	
Code of course:			
Course leader:	dr hab. inż. Włodzimierz Tylus, prof. PWr		
Faculty of the course leader:	W3 Faculty of Chemistry		
Email address of the course leader:	Wlodzimierz.tylus@pwr.edu.pl		
Scientific discipline(s) assigned to	Architecture and urban planning		
the course (doctoral students representing the marked	Automation, electronic, electrical engineering and		
disciplines can participate in the	space technologies		
course):	Information and communication technology		
	Biomedical engineering		
	Chemical engineering		
	Civil engineering, geodesy and transport		
	Materials engineering		
	Mechanical engineering	X	
	Environmental engineering, mining, and energy		
	Mathematics		
	Chemical sciences		
	Physical sciences		
	Management and quality studies		

#### 2. Objectives

- C1 To understand the nature of solid surface in nanotechnology
- C2 Introduction to modern techniques of solid surface testing: XPS/AES, XRD, SEM, TEM, EIS, microhardness/nanohardness, adhesion of coatings and thin films, surface topography, contact profilometry
- C3 Understand the interactions of the surface of the material under study with the corrosive environment. Familiarise with modern laboratory methods of investigating corrosion processes.
- C4 To get acquainted with and use rules in measurements.
- C5 To provide an understanding of physical principles of coherent and incoherent scattering
- C6, C7 To provide an overview of experimental techniques in X-ray diffraction
- C8 to provide practical examples of X-ray scattering experiments and methods of scattering data treatment



## 3. Content

Detailed information about the course content, including topics and form of classes.

No.	Торіс	Number of hours	Form of classes
1	Photoelectric process - primary and secondary emission. Basic concepts: nano-surface, spectroscopic and X-ray notation. X-ray photoelectron spectroscopy (XPS/ESCA). Auger electron spectroscopy (AES). 'Depth' in XPS/AES analyses.	2	lecture
2	Widmo XPS/AES i jego składowe. Etapy procesu analitycznego. Podstawowe instrumentarium (UHV, analizator energii, źródło fotonów, działo jonowe, manipulator). Techniki przygotowania próbek	2	lecture
3	Application of electron spectroscopy in materials engineering. Examples of spectra (XPS/AES) and their interpretation: in microelectronics, semiconductor and polymeric materials, metallurgy, corrosion of materials, ceramics, catalysis	2	lecture
4	Depth profiling (destructive and non-destructive methods). Surface nanomorphology in XPS studies. 3D models. Typical problems in XPS research. Use of XPS databases.	2	lecture
5	Principles of X-ray scattering and diffraction. Basic properties and sources of X-rays. Elastic X-ray scattering by an atom. Scattering from an atomic lattice. Interference and Bragg diffraction. Concept of the Bravais' lattice. Laue interference function. Reciprocal lattice and Ewald's sphere.	2	lecture
6	Scattering and diffraction experiments. Conventional crystallographic experiment and four-circle diffractometer. Experiments with polycrystalline samples. Isotropic vs oriented samples. Fiber diffraction and Polanyi experiment. Short introduction to discovery of DNA structure.	2	lecture
7	Small-angle X-ray scattering. Babinet principle. Scattering power laws for dilute dispersions. Scattering invariant and interface area. Guinier approximation and dimensionality. Small angle scattering from condensed systems. Correlation function.	2	lecture
8	Case studies. 1) Determination of crystal structure of a polycrystalline sample from a powder pattern 2) determination of phase composition of a semicrystalline polymer 3) Determination of particle shape and dimensions from a small-angle scattering profile of a nanocomposite.	2	lecture
9	Basics of electron microscopy (SEM,TEM) and X-ray microanalysis (EDS, WDS). Instruments used: electron	2	lecture



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and X-ray detectors, vacuum system. Preparations used in electron microscopy and their preparation.		
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Application of electron microscopy (SEM, TEM) and	2	lecture
microanalysis of chemical composition (EDS, WDS) in		
material engineering. Study of the structure of		
materials by the method of diffraction of		
backscattered electrons. Examples of photos (SEM,		
TEM), spectra and chemical composition (EDS, WDS)		
and maps of crystallographic orientation (EBSD).		
Basics of interpretation of measurement data.		
Scanning transmission electron microscopy (STEM) -	2	lecture
advantages and disadvantages compared to		
conventional SEM imaging. Concentrated ion beam		
systems and DualBeam ™ systems.		
Polarization measurements in the determination of	2	laboratory
corrosion resistance of construction and special		
materials. Basic concepts, techniques, equipment and		
data interpretation.		
Electrochemical impedance spectroscopy (EIS) in	2	lecture
corrosion studies. Basic concepts, impedance and		
methods of its presentation, available measurement		
modes and electrical equivalent models.		
Electrochemical impedance spectroscopy (EIS) in	2	laboratory
corrosion studies. Measuring system, measurement,		
fitting of the impedance spectra, interpretation.		
Determination of microhardness and adhesion of	2	laboratory
materials, coatings and thin layers.		
	microanalysis of chemical composition (EDS, WDS) in material engineering. Study of the structure of materials by the method of diffraction of backscattered electrons. Examples of photos (SEM, TEM), spectra and chemical composition (EDS, WDS) and maps of crystallographic orientation (EBSD). Basics of interpretation of measurement data. Scanning transmission electron microscopy (STEM) - advantages and disadvantages compared to conventional SEM imaging. Concentrated ion beam systems and DualBeam ™ systems. Polarization measurements in the determination of corrosion resistance of construction and special materials. Basic concepts, techniques, equipment and data interpretation. Electrochemical impedance spectroscopy (EIS) in corrosion studies. Basic concepts, impedance and methods of its presentation, available measurement modes and electrical equivalent models. Electrochemical impedance spectroscopy (EIS) in corrosion studies. Measuring system, measurement, fitting of the impedance spectra, interpretation. Determination of microhardness and adhesion of	microanalysis of chemical composition (EDS, WDS) in material engineering. Study of the structure of materials by the method of diffraction of backscattered electrons. Examples of photos (SEM, TEM), spectra and chemical composition (EDS, WDS) and maps of crystallographic orientation (EBSD). Basics of interpretation of measurement data. Scanning transmission electron microscopy (STEM) - advantages and disadvantages compared to conventional SEM imaging. Concentrated ion beam systems and DualBeam ™ systems. Polarization measurements in the determination of corrosion resistance of construction and special materials. Basic concepts, techniques, equipment and data interpretation. Electrochemical impedance spectroscopy (EIS) in corrosion studies. Basic concepts, impedance and methods of its presentation, available measurement modes and electrical equivalent models. Electrochemical impedance spectroscopy (EIS) in corrosion studies. Measuring system, measurement, fitting of the impedance spectra, interpretation. Determination of microhardness and adhesion of 2

#### 4. Prerequisites

List of prerequisites relating to knowledge, skills and other competences for course participants.

- 1. basic knowledge of atomic structure and chemical bonding
- 2. basic knowledge of electromagnetic waves physics
- 3. basic understanding of crystal structure

#### 5. Learning outcomes

*List of learning outcomes at level 8 of the Polish Qualifications Framework assigned to the course (mark the learning outcomes in the last column).* 

Symbol	Learning outcome	
	KNOWLEDGE. Doctoral student knows and understands:	
SzD_W3	the main trends in the development of the scientific or artistic disciplines covered	$\boxtimes$
	in the curricula;	
SzD_W4	research methodology;	$\boxtimes$



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SzD_W5	the rules for the dissemination of scientific results, including in open access mode;	
SzD_W6	the fundamental dilemmas of modern civilization;	
SzD_W7	the legal and ethical conditions of scientific activity;	
SzD_W8	the economic and other relevant conditions of scientific activity;	
SzD_W9	basic principles of knowledge transfer to the economic and social spheres and	
	commercialisation of results of scientific activity and know-how related to these	
	results.	
-	SKILLS. Doctoral student is able to:	
SzD_U2	<ul> <li>use knowledge from different fields of science or art to creatively identify,</li> <li>formulate and innovatively solve complex problems or perform research tasks, in</li> <li>particular:</li> <li>define the purpose and subject of scientific research, formulate a research hypothesis,</li> </ul>	
	<ul> <li>develop research methods, techniques and tools, and use them creatively,</li> <li>draw conclusions on the basis of scientific research;</li> <li>critically analyse and evaluate the results of scientific research, expertise and</li> <li>other creative work and their contribution to knowledge development;</li> <li>transfer the results of scientific activities to the economic and social spheres;</li> </ul>	
SzD_U3	communicate on specialised topics to the extent that they enable an active participation in the international scientific community;	$\boxtimes$
SzD_U4	disseminate research results, including in popular forms;	
SzD_U5	initiate debates and participate in a scientific discourse;	
SzD_U6	be able to speak a foreign language at B2 level of the Common European Framework of Reference for Languages to a level that enables them to participate in the international scientific and professional environment;	
SzD_U7	plan and implement an individual or collective research or creative activity, including in an international environment;	
SzD_U8	independently plan and act for one's own development and inspire and organize the development of others;	
SzD_U9	plan classes or groups of classes and implement them using modern methods and tools.	
	SOCIAL COMPETENCES. Doctoral student is ready to:	
SzD_K3	fulfilling the social obligations of researchers and creators, initiate public interest activities, thinking and acting in an entrepreneurial way;	
SzD_K4	<ul> <li>maintaining and developing the ethos of research and creative environments, including:</li> <li>carrying out scientific activities in an independent manner,</li> <li>respecting the principle of public ownership of research results, taking into account the principles of intellectual property protection.</li> </ul>	

### 6. Evaluation

Short description of the method(s) used to evaluate the learning outcomes assigned to the course, e.g., exam, test, report, presentation, etc.



#### 7. Teaching methods

Short description of the teaching methods used during the course, e.g., multimedia presentation, discussion, literature studies, developing written documents, own work, etc.

Author's lecture combined with a laboratory demonstration, discussion, literature studies

#### 8. Literature

*List of primary and secondary literature used to prepare the course and including additional knowledge for participants, e.g., books, textbooks, research papers, standards, web pages, etc.* 

- 1. B.E. Warren "X-ray diffraction" Courier Corporation, 1990
- 2. N. Stribeck "X-ray scattering of soft matter" Springer-Verlag Berlin-Heidelberg 2007
- 3. An Introducing to Surface Analysis by XPS and AES; J.F. Watts, J.Wolstenholme, John Wiley&Sons Ltd., 2003
- 4. Scanning Electrochemical Microscopy, Second Edition; Allen J. Bard, Michael V. Mirkin, CRC Press, 2012.
- 5. Kędzierski Z., Stępiński J.: Elektronowy mikroskop skaningowy (SEM). UWN AGH, 2007
- 6. W.C. Oliver and G.M. Pharr, An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments, Journal of Materials Research, Volume 7, Issue 6 June 1992, pp. 1564-1583.
- 7. Ian N. Sneddon, The relation between load and penetration in the axisymmetric boussinesq problem for a punch of arbitrary profile, International Journal of Engineering Science, Volume 3, Issue 1, May 1965, Pages 47-57.
- 8. EN 1071-3; Advanced technical ceramics –Methods of test for ceramic coatings -Part 3: Determination of adhesion and other mechanical failure modes by a scratch test.
- 9. Standard Test Method for Adhesion Strength and Mechanical Failure Modes of Ceramic Coatings by Quantitative Single Point Scratch Testing, C1624 05 (Reapproved 2015) ASTM.
- 10. https://www.gamry.com/
- 11. A. Lasia, Electrochemical Impedance Spectroscopy and its Applications, © Springer Science+Business Media New York 2014.
- 12. CasaXPS Manual 2.3.15 Rev 1.2, Intrudction to XPS and AES, N. Fairley, Casa Software Ltd, <a href="http://www.casaxps.com/ebooks/ebooks.htm">http://www.casaxps.com/ebooks/ebooks.htm</a>

#### 9. Other remarks

Additional remarks, comments, (e.g., language of the course)