



**Projekt Nr WND-POWR.03.02.00-00-I043/16**

**Międzynarodowe interdyscyplinarne studia doktoranckie z zakresu nauk o materiałach z wykładowym językiem angielskim**

**Program Operacyjny Wiedza Edukacja Rozwój 2014-2020,**

**Działanie 3.2 Studia doktoranckie**

**Program Studiów**

**Project WND-POWR.03.02.00-00-I043/16**

**International interdisciplinary PhD Studies in Materials Science with English as the language of instruction**

**The program of the PhD Studies**

**Prof. MAREK FARYNA PhD, DSc**

**Head of PhD Studies**

**Krakow, September 2017**

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*Program Operacyjny Wiedza Edukacja Rozwój 2014-2020, Działanie 3.2 Studia doktoranckie*

## Introduction

The PhD studies are organized in four main areas, i.e.:

### 1. Environmental-friendly materials and technologies

- Lead-free solders
- Multicrystalline silicon solar cells
- Biocompatible coating in blood contacting materials

### 2. Knowledge-based multifunctional materials

- Gradient materials produced using different methods
- Light alloys of new generation with improved mechanical properties
- Production and optimization of intermetallic properties
- Bulk metallic glasses

### 3. Nano- and microcrystalline materials

- Mechanical alloying and hot-pressing of intermetallics
- Severe plastic deformation and fabrication of ultra-fine grain materials

### 4. Development of modern research tools and diagnostic methods

- Crystallographic orientation mapping in respect to diagnosis and prognosis of mechanical properties of metallic, ceramic and composite materials based on the scanning and transmission electron microscopy examinations of local grain orientations
- Data processing of local crystallographic orientations; orientation distribution function, orientation topography, quantitative description of microstructure
- Complex characteristics of advanced materials using new transmission electron microscopy techniques.

The above fields are only roughly defined. Each member of the Institute scientific staff works in at least two of these fields and a number of subjects appear simultaneously on the lists of research subjects. There is a great deal of interaction between the fields.

Students are expected to learn fundamentals of their chosen field and to develop a deep understanding of one their significant aspects. Students are required to take further subjects designated by their academic advisor. A full range of advanced-level subjects is offered in each field, and arrangements can be made for individually planned study of any topic. Oral

examinations in the academic program for the doctoral degree are designed accordingly. Participation in all Institute seminars is obligatory.

Presently, a large research program on the structure and properties, preparation, and processing of materials, with emphasis on ceramics, metals and biomaterials, is conducted in the Institute. Students choose research projects from several possibilities that exist within the Institute and work closely with its scientific supervisor. The results of the thesis must be of sufficient significance to warrant publication in the scientific periodicals.

The proposed program is based on a novel approach taking into account the current needs indispensable for the PhD students while completing their thesis in the field of materials science. It includes not only the lectures which cover the knowledge of newly developed materials and methods of their characterization but also soft skills such as acquiring financial resources for conducting research or how to write scientific papers and successfully prepare PhD thesis.

The PhD studies has a stationary character, are free of charge (FOC) and substantially financially supported by European Community.

The PhD studies has an interdisciplinary character as they cover a wide range of topics in the field of materials science, starting from biomaterials and finishing to solar cell systems.

International character and mobility of the PhD studies will be provided by the broad scientific exchange with German partners i.e. Dresden Center of Nanoanalysis (DCN), Technische Universität Dresden and Fraunhofer–Institut für Keramische Technologies and Systeme (IKTS), Dresden.

The PhD studies will familiarize participants with global experience in the commercialization of scientific research and promote open attitudes to science and business cooperation and readiness for commercialization of scientific knowledge.

Each participant will gain access to develop individual skills during scientific seminars at which the participants will share their knowledge and discuss the problems they encounter.

The Institute of Metallurgy and Materials Science has a number of well-equipped research laboratories. There is a close interaction between them including the sharing of experimental facilities and equipment. Most of experimental facilities are extensively used in the frame of Testing Laboratories authorized by Polish Centre for Testing and Certification in accordance



with ISO standards. The certificate of conformance with Polish and European standards PN-ISO/IEC 17025:2001 for testing methods in the range of mechanical and structural properties of metals and alloys is valid till the next audit in 2019.

## Year 1

### Semester I

Course: **Advanced scanning electron microscopy in materials science**  
**(Techniki analityczne w skaningowej mikroskopii elektronowej)**

(15 hours, exam, 1 ECTS)

#### *Course description*

##### **1. Electron Beam –Specimen Interaction (part I)**

scope: Elastic and inelastic scattering, interaction volume, Monte Carlo simulation, electron range.

##### **2. Electron Beam Specimen Interaction (part II)**

scope: Imaging signals from interaction volume (backscatter electrons, secondary electrons).

##### **3. Scanning Electron Microscope (part I)**

scope: Introductory remarks about spatial resolution and depth of field (focus), electron probe diameter versus electron current, how the SEM works, electron guns and their characteristics.

##### **4. Scanning Electron Microscope (part II)**

scope: Electron optics, lenses and their aberrations, electron detectors, the role of specimen and detectors in contrast formation.

##### **5. Energy Dispersive Spectrometry**

scope: Generation of X-Rays production, continuum X-Ray production (Brehmsstrahlung), characteristic X-Ray production, depth of X-Ray production, X-Ray absorption, X-Ray Fluorescence, Energy dispersive X-ray Spectrometer - operating principles, detection process, artefacts.

##### **6. Wavelength Dispersive Spectrometry**

scope: Introduction, basic principles, diffraction conditions, diffraction crystals, X-ray proportional counter, comparison of Wavelength Dispersive Spectrometers with Conventional Energy Dispersive Spectrometers.

##### **7. Quantitative X-ray Microanalysis**

scope: Introduction, Quantitative analysis procedures, the approach to X-Ray Quantification: the need of matrix correction, the physical origin of matrix effects, ZAF factors in Microanalysis, calculation of ZAF factors, practical aspects.

### **8. Variable Pressure/Environmental Scanning Electron Microscopy**

scope: General principles of VP-SEM: utilizing a gas, imaging and analysis in VP-SEM: the influence of a gas, imaging uncoated specimens in the VP-SEM, X-Ray microanalysis in low vacuum conditions.

### **9. Electron Backscatter Diffraction (part I)**

scope: Theoretical framework for electron backscatter diffraction, fundamentals of automated EBSD, the influence of microstructure and SEM settings on quality of diffraction pattern, phase identification.

### **10. Electron Backscatter Diffraction (part II)**

scope: Advanced software capabilities for automated EBSD, EBSD from non-conductive specimens, special EBSD techniques: 3 dimensional EBSD, EBSD at elevated temperatures.

### ***Course is based on the following literature:***

- Scanning Electron Microscopy and X-Ray Microanalysis (Third Edition), Joseph Goldstein, Dale Newbury, David Joy, Charles Lyman, Patrick Echlin, Eric Lifshin, Linda Sawyer and Joseph Michael, Kluwer Academics/Plenum Publishers, 2003
  - Electron Microscopy and Analysis, (Third Edition), Peter Goodhew, John Humphries, Richard Beanland, Taylor & Francis, London, 2001
  - Electron Microprobe Analysis, (Second Edition), S.J.B. Reed, Cambridge University Press, 1993
  - Electron Probe Quantification, K.F.J. Heinrich and D.E. Newbury, Plenum Press, New York, 1991
  - Principles and Practice of Variable Pressure/Environmental Scanning Electron Microscopy, Debbie Stokes, John Wiley & Sons, 2008
- .....

## Course: **Acquiring financial resources for conducting research**

### **Pozyskiwanie środków finansowych na prowadzenie badań naukowych**

(10h, exam, 2 ECTS)

#### ***Course summary***

The course aims at acquainting the participants with the objectives of the Europe 2020 initiative and the national program documents aimed at supporting innovation and R & D. Participants will learn the mechanisms for applying for national and international research support instruments. Classes will be conducted in a course and workshop. Participants will gain knowledge about institutions supporting R & D projects, as well as skills in developing application documentation.

#### ***Course description***

1. Scientific research in EU policy.
2. National R & D support program.
3. International R & D programs (HORIZON 2020)
4. National R & D Support Programs (National Science Center, National Center for Research and Development, National and Regional Operational Programs 2014-2020)
5. Principles of preparation of application documentation.
6. R & D projects targeted for implementation
7. Institutions supporting the preparation of R & D projects

#### ***Course is based on the following literature:***

- Strategic opinion for research and innovation in the HORIZON 2020 work programme 2018 – 2020
- Stakeholder Consultation on the Strategy for Research and Innovation activities in the HORIZON 2020 Work Programme for Nanotechnologies, Advanced Materials, Biotechnology, and Advanced Manufacturing and Processing (NMBP)
- W. M. Grudzewski, I. K. Hejduk, „Zarządzanie technologiami. Zaawansowane technologie i wyzwanie ich komercjalizacji” Wyd. Difin, 2008
- D. Francis, „Developing Innovative Capability”, University of Brighton, Brighton 2001

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- B. Godin, “The Politics of Innovation: Machiavelli and Political Innovation, or, How to Stabilize a Changing World”, Project on the Intellectual History of Innovation, Working Paper No. 17, 2014

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### **Scientific Seminar**

(10 h, credit, 1 ECTS)

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### **Consultations with Supervisor**

(2 h, credit, 1 ECTS)





## Semester II

Course: **Materials: fundamentals, engineering, processing, design**  
(**Materialy: podstawy naukowe, inżynieria, procesy, projektowanie**)

(15 h, exam, 2 ECTS)

### *Course summary*

The Course includes an introduction to materials science and engineering focused on science-led approach however it gives little emphasis to design-led. Guiding learning on materials and their structure and properties, crystallography, phase diagrams and phase transformations, processing, diagnostics and application is given. Some information are presented on fundamentals and understanding, control of properties at a different scale as well as materials selection and design. The Course is divided into parts comprising: a basing knowledge, possible application and diagnostics together with examples of chosen experimental results. The Course is dedicated to students motivating their understanding of the nature of modern material design and developing skills.

### *Course description*

1. Engineering materials
2. Atomic bonding and crystallography
3. Mechanical properties
4. Crystal defects of crystalline structure
5. Phase diagrams
6. Structure changes
7. Metals and alloys
8. Ceramic materials and glasses
9. Polymers
10. Composites
11. Intermetallics
12. Amorphous and nanocrystalline materials
13. Porous materials
14. Smart materials
15. Biomaterials

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16. Processing of metals, ceramics, polymers, composites
17. Surface engineering
18. Nanomaterials and nanotechnologies
19. Basics for materials design

***Course is based on the following literature:***

- M.Ashby: Materials; engineering, science, processing and design, Elsevier 2010
- R.Pampuch: ABC of Contemporary Ceramic Materials, Techna Group, 2008
- M.Blicharski: Wstęp do inżynierii materiałowej, Wyd. Nauk.-Techn. 2003
- L.A.Dobrzański: Metalowe materiały inżynierskie, Wyd. Nauk.-Techn., 2004
- Mazurkiewicz: Nanonauki i Nanotechnologie, Wyd.Inst.Technol.Ekspl., Radom 2007

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Course: **Characterization of materials structure by X-ray diffraction techniques**

**(Charakterystyka struktury materiałów techniką dyfrakcji rentgenowskiej)**

(15 h, exam, 2 ECTS)

***Course description***

**1. Nature and sources of the X-rays**

Natural sources, inducing, X-ray tubes, synchrotrons, characteristic and fluorescent radiation, absorption effect.

**2. Diffraction phenomenon of X-ray. Part I**

Diffraction phenomenon and related physical/geometrical laws, diffraction on crystal lattices. Laue equations, intensity of diffracted beam, theories of diffraction, Bormann effect, polarization.

**3. Diffraction phenomenon of X-ray. Part II**

Elementary cells of crystallographic lattice, crystallographic indexing, reciprocal lattice and interpretation of diffraction effects, detection techniques, position-sensitive detection technique, Si-strip detector.

**4. Crystallography and diffraction**

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Symmetry in the nature, Basic definitions in applied crystallography, stereographic projection, pole figures.

### **5. Crystallographic texture. Part I**

Crystallographic orientation, texture components, texture analysis, orientation distribution function and its interpretation.

### **6. Crystallographic texture. Part II**

Modern quantitative texture analysis, calculation of orientation distribution function, demonstration of the *LaboTex* software, examples and practical remarks.

### **7. Texture analysis of polycrystalline materials and X-Ray Texture Tomography**

Metals, polymers, rocks, bio-materials, fatigue wear, effects of changing deformation router, investigations of metals after severe plastic deformation, EBSD, topography of texture.

Texture inhomogeneity, X-Ray Texture Tomography – principles and application.

### **8. Using X-ray diffraction in materials engineering**

Methods of registration the diffraction effects (modes:  $\theta-2\theta$ ,  $\omega-2\theta$ ,  $\omega$ ,  $2\theta$ ), WAS, SAXS, phase transformation monitored by high/low temperature attachments, high-resolution X-ray diffractometry, perfectness of crystal, *Laue- and Debye 'a-Scherr* patterns, indexing the X-ray pattern.

### **9. X-ray phase analysis**

Line profile analysis (programme *DAMfit*), identification of superstructure, X-ray phase analysis (qualitative and quantitative), texture in X-ray quantitative analysis, structure refinement by Rietveld method.

### **10. Other useful methods and the newest achievements in the field of X-ray diffraction**

Estimation of stacking fault energy by X-ray diffraction technique, stress analysis, size of crystallites and lattice distortions, future of X-ray diffraction: free electron laser and high-energy photon beams.

### **11. Demonstration of the X-ray Laboratory and a final colloquium**

Demonstration of measurement procedures, data acquisition and data processing. Examples.



***Course is based on the following literature:***

- Chateigner D. (2006), Combined Analysis: structure-texture-microstructure-phase-stresses-reflectivity determination by x-ray and neutron scattering, CRISMAT-ENSICAEN, UMR CNRS n<sup>o</sup>6508, 6Bd. M.Juin, F-14050 Caen, France
- Mittemeijer E.J., Scardi P. (Eds.), (2004) Diffraction Analysis of the Microstructure of Materials, Springer-Verlag Berlin Heidelberg 2004
- Bojarski, Z., Łągiewka, E.(1988). Rentgenowska analiza strukturalna, PWN, Warszawa.
- Bonarski, J.(2001). Rentgenowska Tomografia Teksturowa, IMIM PAN, Kraków.
- Bunge, H.J.(1982). Texture Analysis in Materials Science. Mathematical Methods. Butterworths Publ. London.
- LaboTex.(2000). The Texture Analysis Software.by LaboSoft s.c.
- Luger, P.(1989). Rentgenografia strukturalna monokryształów. PWN Warszawa
- Przedmojski, J.(1990). Rentgenowskie metody badawcze w inżynierii materiałowej, WNT. Warszawa.
- Sonin, A.S.(1982). O krystalografii, PWN, Warszawa.

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Course: **Phase equilibrium with elements of chemical thermodynamics.**

**(Równowagi fazowe z elementami termodynamiki chemicznej)**

(15 h, exam, 2 ECTS)

***Course description***

**1. The laws of thermodynamics**

Definition of state functions and state parameters

Internal energy, enthalpy, entropy, free energy, free enthalpy

Calculation of the thermodynamic function of the component

**2. Solutions. Classification. Measurement. Interpretation**

Configuration entropy,

Mixing functions

Activity of the component in solution

Classification of solutions

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Measurements of thermodynamic properties

Methods of interpretation of test results

Calculation of partial thermodynamic functions, Gibbs-Duhem equation

### **3. Equilibrium systems. Thermodynamic analysis**

Systems with unlimited solubility

Systems with eutectic and eutectoid, peritropic and peritectoid transformation

Systems with intermetallic phases

Systems with a miscibility gap in the liquid and solid state

Spinodal transformation systems

Equilibrium metal (solution) - vapour

Calculation of phase equilibria.

### **4. Calculation of the of free energy change of reaction. Equilibrium constant.**

*Course is based on the following literature:*

- 1. Chiranjib Kumar Gupta, Chemical Metallurgy, Wiley-VCH GmbH & Co. KGaA, 2003
- 2. J.J. Moor, Chemical Metallurgy, Butterworth & Heinemann Ltd 1990
- 3. K. Gumiński, Termodynamika, Warszawa 1982, PWN
- 4. Praca zbiorowa, Chemia fizyczna, Warszawa 1980, PWN
- 5. J. Kaczyński, S. Prowans, Podstawy teoretyczne metaloznawstwa, Wydawnictwo Śląsk 1972

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### **Scientific Seminar**

(10 h, credit, 1 ECTS)

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### **Consultations with Supervisor**

(2 h, credit, 1 ECTS)

## Year 2

### Semester I

Course: **Solidification of alloys for special purposes**  
**(Krystalizacja stopów dla specjalnych zastosowań)**

(15 h, exam, 2 ECTS)

#### *Course description*

##### **1. Fundamentals of solidification**

Description of typical structures appeared in the massive ingot.

Structure formation under positive and negative thermal gradients.

Space-time-structure map for the massive steel/cast iron roll as it results from the temperature field analysis.

Columnar  $\rightleftharpoons$  equiaxed structure transition (CET) due to the thermal gradient field calculated numerically for the solidification of massive ingot.

Scheil's theory for the non-diffusive non-equilibrium solidification/micro-segregation.

Equilibrium solidification as it results from the mass balance (so-called Lever Rule).

New theory for solidification based on two phenomena: solute partitioning and solute redistribution after back-diffusion.

Perfect mathematical reduction of the new theory to the Scheil's model and to the equilibrium solidification.

Development of the Scheil's theory for the multi-peritectic systems and multi-peritectic/eutectic systems.

Principle of unidirectional solidification – the Bridgman's system

##### **2. Theory of diffusion soldering/brazing**

Description of phenomena which occur during soldering/brazing like: dissolution, solidification, solid/solid transformation.

Diffusion zones within the substrate.

Application of the Umeda-Okane-Kurz criterion to justify the occurrence of technology under meta-stable conditions.

Application of the new theory for solidification based on partitioning and solute redistribution after back-diffusion and accompanied by the undercooled peritectic reactions.

Development of the new theory for the multi-peritectic systems and multi-peritectic/eutectic systems.

Calculations of the phase diagrams for the meta-stable equilibrium (Thermocalc Software): a/ for dissolution, b/ for solidification accompanied by the peritectic reactions resulting in the intermetallic phases/compounds formation.

Experimental justification for the non-influence of time and non-influence of real temperature on the average solute concentration within the interconnection.

Determination of the solidification path, solid/liquid interface path and solute redistribution path for the diffusion soldering/brazing.

Simulation of the diffusion joint formation (reproduction of a ratio of the sub-layers thicknesses and the solute concentration profiles across the given joint sub-layers).

Mass balance within the diffusion interconnection.

### **3. Model for the solute micro-field ahead of the solid/liquid interface of a growing lamellar eutectic**

Improvement of the Jackson-Hunt's theory for the lamellar eutectic growth.

Replacement of the ideally coupled growth by the coupled growth with differentiated undercooling of both eutectic phases.

New solution to differential diffusion equation.

New boundary condition for the solution to diffusion equation.

Localization of mechanical equilibrium, thermodynamic equilibrium and protrusion of the leading eutectic phase over the wetting eutectic phase.

Application of the calculation of the entropy production due to the new description of the solid/liquid interface.

Total mass balance and local mass balance.

The relationship between growth rate and protrusion.

### **4. Theory for the lamella → rod transformation in some eutectic alloys**

Critical discussion of the Jackson-Hunt's theory for the prediction of the lamellar or rod-like structure formation within the eutectic alloys.

Model for the irregular eutectic structure formation based on both a/ criterion of the entropy production minimum and b/ concept of the marginal stability.

Transformation irregular → regular eutectic structure shown on the paraboloid of entropy production on which trajectory of local minima of entropy production for stationary states and trajectory of marginal stability are drawn schematically.

Oscillation of the structure parameters.

Growth laws for the lamellar structure formation and for the rod-like structure formation of regular eutectics developed due to the application of the criterion of the minimum entropy production.

Experimental determination the threshold rate and operating range of growth rates for the lamella → rod transformation of the Al-Si eutectic.

Simulation of the lamella → rod transformation by the selection of lower minimum of entropy production (minimum at which rod-like structure formation occurs or minimum of entropy production at which lamellar structure formation is observed).

***Course is based on the following literature:***

- W. Kurz, J.D. Fisher, Fundamentals of Solidification, Trans Tech Publications – book
- Prigogine, Introduction a la Thermodynamique des Processus Irreversible – book
- W. Wołczyński, Lectures via Internet: METallurgical TRaining Online (METRO)
  - Mass transport at the solid/liquid interface of growing composite *in situ*
  - Transformation: lamella – rod within oriented eutectic Al-Si
  - Solidification / microsegregation model applied to description of diffusion soldering / brazing
- G. Lesoult, M. Turpin, Etude Theorique sur la Croissance des Eutectiques Lamellaires, *Revue Scientifique de la Revue de Metallurgie*, Vol. 66, (1969). pp. 619-631
- E. Scheil, Über die Eutektische Kristallisation, *Zeitschrift für Metallkunde*, Vol. 34, (1942), pp. 70-80
- W. Wołczyński, Thermodynamics of Irregular Eutectic Growth, *Materials Science Forum*, Vol. 215/216, (1996), pp. 303-312
- W. Wołczyński, Back-Diffusion Phenomenon during the Crystal Growth by the Bridgman Method, In: *Modelling of Transport Phenomena in Crystal Growth*, J.S.

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Szmyd & K. Suzuki, (Ed.), pp. 19-59, WIT PRESS ISBN: 1-85312-735-3, Ashurst Lodge, Southampton, UK - Boston, USA (2000)

- W. Wołczyński, Concentration Micro-Field for Lamellar Eutectic Growth, *Defect and Diffusion Forum*, Vol. 272, (2007), pp. 123-138
- W. Wołczyński, Lamella / Rod Transformation as described by the Criterion of the Minimum Entropy Production, *International Journal of Thermodynamics*, Vol. 13, (2010), pp. 35-42

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**Course: Fundamentals of Thermal Analysis and Differential Scanning Calorimetry Application in Materials Science Investigations**  
**(Analiza cieplna i kalorymetria różnicowa w badaniach materiałów)**

(15 h, exam, 2 ECTS)

***Course summary:***

The course will include theory and practical application of the thermal analysis and calorimetry methods most often required for different materials' and processes' characterisation in materials science. The course will be concentrated on metallic materials and related phase transformations. Nevertheless, other types of materials like chemical compounds and macromolecular materials are included. The course covers differential thermal analysis (DTA), differential scanning, pressure and temperature differential modulated calorimetry (DSC, PDSC, MDSC), thermal gravimetry (TGA, DTGA) and thermo-mechanical analysis (TMA) both in the aspect of spontaneous dimensional changes and elastic/plastic deformation under different loading. Constant and modulated loads' application (DTMA) will be discussed. Examples of complex characterisation by the application of the complementary methods like DSC/TMA and TA/DSC/TGA will be presented. Fundamental laws of equilibrium thermodynamics, simple phase diagrams' reading and phase transformation kinetics in relation to the thermal analysis and calorimetry results interpretation are included.

***Course description:***

**Introduction**

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1. Heat-heat transfer- thermal equilibrium-irreversible transformations;
2. What is heat: historical development of the idea, temperature measurements, historical development of thermal measurements; modern technology;
3. Revision: laws of thermodynamics, equilibrium/ non equilibrium thermodynamics in relation to thermal analysis with heating rates commonly applied in calorimetry.
4. Revision: phase equilibria – basic types of phase diagrams - crystallization/precipitation /dissolution processes proceeding with average and high heating rates;
5. Recent developments in methods of thermal analysis and calorimetry;

### **High temperature thermal analysis and calorimetry**

6. DTA, TGA, DSC -method classification, advantages, similarities and differences; basic construction of the equipment, underlying mathematical description. Calorimetry- from isothermal to reactive calorimetry, different ideas and construction of calorimeters, DSC- how it qualifies in relation to “classic” calorimetry;
7. DTA- high temperature differential analysis, construction of commercially available equipment, examples of scientific investigation ; DTA application for the phase diagram verification.
8. TGA and TGA-DSC methods; equipment construction, different modifications, advantages; examples of application, oxidation studies;
9. DTA, TGA, high temperature DSC: scientific and industrial applications for materials’ characterisation and quality testing: from cements to energy materials.
10. Practical presentation of the DTA, high temperature DSC and SDT equipment, methods and interpretation in the Laboratory of Thermal Analysis and Calorimetry.

### **Precise calorimetry at the average and low temperatures**

11. Construction of the most common DSC calorimeters, simplified and developed calorimetric equations. Typical applications of DSC technique in materials sciences;
12. DSC development: extremely and high rate, modulated and pressure DSC, micro-DSC, application;
13. The meaning of heat capacity, the Ehrenfest classification of phase transitions, the DSC and MDSC application in Cp determination;

14. Basic description of transformation kinetic, kinetic models and model free kinetic, common solutions supplied by the producers;
15. Practical presentation of the DSC, MDSC and PDSC equipment, methods and interpretation in the Laboratory of Thermal Analysis and Calorimetry;

**Thermal analysis basing on dimensional changes with temperature and load**

16. TMA analysis and Dilatometry - typical instrument construction and properties; basic classification of the mechanical behaviour of the continuous solid state;
17. Elasto-plastic properties of chosen materials; application of different TMA and DTMA modes of operation in material investigation, testing and qualification;
18. The application of complementary thermal analysis methods;
19. Practical presentation of the TMA equipment, methods and interpretation in the Laboratory of Thermal Analysis and Calorimetry.

***The course is based on the following literature:***

1. Wojciech Zielenkiewicz, "Calorimetry", second edition 2008, Institute of Physical Chemistry of the Polish Academy of Sciences
2. B. Wunderlich, "Thermal Analysis", 1990, Academic Press Inc., Harcourt, Brace, Jovanovich Publishers
3. G.W.H. Höhne, W. Hemminger, H-J. Flammersheim, "Differential Scanning Calorimetry. An Introduction for Practitioners", 1996, Springer-Verlag Berlin, Heidelberg
4. "Thermal Analysis in Metallurgy" Ed. R.D. Shull and A. Joshi, 1990, TMS;
5. "Glassy, amorphous and Nano-crystalline Materials" , Series: "Hot Topics in Thermal Analysis and Calorimetry" eds. J. Šesták, JJ. Mareš, P. Hubík, series ed. J. Simon, Springer, London, Heidelberg, New York
6. Current scientific publications
7. Equipment technical notes TAI, Netzsch comp. Mettler-Toledo, Manuals of TAI and Netzsch Comp.
8. Periodic publications: -"Thermal Analysis, Information for Users" UserCom, Mettler-Toledo
9. Zbigniew Kędzierski, "Termodynamika Stopów", 1999, AGH, Uczelniane Wydawnictwo Naukowo-Dydaktyczne, Kraków

10. A.M. Zaharov, „Diagrammy Sostojanij Dwojnych i Trojnych Sistem” 1964, Izdatielstwo Metallurgija (Ros.)
11. W.P. Skripov, B.P. Koverda, „Spontannaja Kristalizacija Pereohlozdiennyh zidkostiej” 1984 Izdatielstwo Nauka (Ros.)
12. Janina Ostrowska-Maciejewska, „Podstawy Mechaniki Ośrodków Ciągłych” 1982 PWN, Warszawa
13. Jerzy Grabarczyk, „Wstęp do fizyki ciała stałego” 2000, Warszawa, Oficyna Wydawnicza Politechniki Warszawskiej

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**Course: How to write scientific papers and prepare PhD thesis?**  
**(Jak pisać prace naukowe oraz jak prawidłowo przygotować rozprawę doktorską?)**

(7 h, credit, 2 ETCS)

***Seminar summary***

The seminar is aimed at discussing different ways of presentation of results of investigations to scientific community. The discussion of final, partial or even preliminary results with other specialists in the field is one of most important thing in reaching the right solution of the analyzed problem. Presently, scientists may communicate through different channels, like seminars, conferences, international congresses, aside from writing papers to scientific journals. There are well defined rules which such communications should fulfill, i.e. they should be divided to parts which enable easier understanding of their content. Additionally, each part should include strictly pre-defined information. However, all these possibilities require significantly or at least slightly different approach. The presentation of results should be clear and as short as possible but simultaneously properly backed with experimental data. The overview of the field justifying both the start of experiment and publishing its results is one of most important thing. The discussion of results needs its verification in papers listed in overview. All this should be well balanced to make a good paper. The specified above concept of sharing results was elaborated to help to understand each other results. Of course, these rules

might be changed in some special case, but such situation should be well documented and explained. Therefore, the present seminar will explain the most important points which should be considered, while writing abstracts, extended abstracts, short communications, and full length papers. Finally, the proper arrangement of Ph. D thesis, i.e. presentation based on only most important results from a study including a four of five years will be proposed.

***The seminar is based on the following literature:***

- Liśkiewicz T., Liśkiewicz G., Wprowadzenie do efektywnego publikowania naukowego. Jak przygotować, wysłać i promować artykuł naukowy. Wyd. AmberEditing, Łódź. 2014.
- Janusz Biernat, Profesjonalna publikacja, Politechnika Wrocławska 2003,
- Michał Żmijewski, Jak napisać dobry artykuł przeglądowy, Uniwersytet Medyczny, Gdańsk 2011.
- Jacek Wytrębowski, O poprawności językowej publikacji naukowo-technicznych, Politechnika Warszawska, ZAGADNIENIA NAUKOZNAWSTWA 1 (179), 2009 PL ISSN 0044 – 1619

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**Scientific Seminar**

(10 h, credit, 1 ECTS)

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**Consultations with Supervisor**

(2 h, credit, 1 ECTS)

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## Semester II

### Course: **Application of advanced transmission electron microscopy techniques in materials research**

### **(Wykorzystanie zaawansowanych metod mikroskopii transmisyjnej w badaniach materiałowych)**

(15 h, exam, 1 ECTS)

#### *Course summary*

The course is divided to several parts, i.e. classical transmission electron microscopy (TEM) techniques, advanced techniques including high resolution and energy filtering, sample preparation. The course will be finish with examples of application of TEM method to advanced materials characterization.

The classical transmission microscopy will cover diffraction and mass-thickness contrast problems. The description of diffraction techniques would include setting microscope for obtaining Selected Area (SA) diffraction, micro-diffraction and Convergent Beam Electron Diffraction (CBED). Next, formation of high resolution images at two beam condition and on axis orientation will be discussed. The part of analytical microscopy will concentrate on EDS systems, i.e. interaction of electron beam with a thin foil, proper condition to acquire EDS spectra, its qualitative and quantitative processing as well as possible artifact. The separate time will be assign to energy filtering techniques including Gatan Image Filtering (GIF). The analytical part will be finished with presentation concerning some special application from that field like Atom Location by Channeling Enhanced Microanalysis (ALCHEMI).

The examples of problem solving with TEM will cover nano-composite CrN/Si<sub>3</sub>N<sub>4</sub> coatings, multilayers of Ni/Al, Ni/Cu and Fr/Cr type as well as bulk Al<sub>x</sub>xxx/Saffil fibers nano-composites. They all were chosen to show a proper way, how to plan such experiments starting from sample preparation stage and finishing on choosing a proper TEM technique.

#### *Course is based on the following literature:*

- R. D. Heidenreich, Fundamentals of Transmission Electron Microscopy
- J.W. Edington, Practical Electron Microscopy in Materials Science
- D. B. Williams and C. B. Carter, Transmission Electron Microscopy

- D.B. Williams, Practical Analytical Electron Microscopy in Materials Science
- G. Thomas, Transmission Electron Microscopy of Metals
- J.H. Spence, J.M. Zuo, Electron Microdiffraction
- I.P. Jones, Chemical Microanalysis Using Electron Beams

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## Course: **Advanced materials for special applications**

### **(Materiały funkcjonalne i konstrukcyjne)**

(15 h, exam, 2 ECTS)

#### ***Course summary:***

The task of the course is to extend the knowledge of students in the area of novel materials that fulfill requirements of recent industrial trends in directions of ecology, particularly contributing to lower fuel consumption due to lower weight of vehicles, higher engine efficiency due to application of better heat resistant materials; furthermore biocompatible materials including new titanium alloys for implants and new nickel free intelligent materials; recycling of materials what support further use of metallic materials. There are also new trends in composites promoting application as a hardening phase of carbonous materials such as graphene platelets, carbon nanotubes and others that allow to increase strength and electrical and thermal conductivity at the same time. Development in the field of metallic materials will include new high entropy alloys, intermetallic materials, application of spinodal decomposition; similarly in the field of ceramic materials attention will be given to intelligent materials, biomaterials and composites.

#### ***Course description***

1. Historical view of constructional materials and summary of carbon steels and alloyed steels in view of development of a new steels of high strength and improved plasticity like bainitic steels, TRIP steels, HSLA and others.
2. Light alloys and new aluminum and magnesium alloys and directions of development such as ultrahigh strength nano-size grain alloys or ultralight MgLi base alloys.
3. Metallic and ceramic biomaterials, historical background and directions of application, particularly Ni free intelligent alloys and Al and V free Ti alloys for bio applications.

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4. Nanomaterials including methods of grain refinement such as bottom up and top down, characterization of mechanical and physical properties; structure studies and examples of current and future application.
5. Composites, production, properties, structure and applications, future trends including application of carbonous materials such graphene and carbon nanotubes for better strength and conductivity.
6. Amorphous materials, manufacturing, characterization, properties and perspectives of application of high strength and ultralight alloys and of iron base of excellent soft magnetic properties.
7. Ceramic materials for high temperature use and ultra hard with good wear properties, new materials with high toughness, thermoelectric materials and composites, their structure, manufacturing and properties.
8. Trends in development of metallic materials like high entropy alloys and intermetallics for high temperature applications, application of spinodal decomposition, magnetic shape memory alloys and others.

***Course is based on the following literature:***

- Marek Blicharski „Inżynieria Materiałowa” wyd. PWN Warszawa 2002
- A.R. Olszyna „Ceramika supertwarda” Oficyna Wydawnicza Politechniki Warszawskiej, Warszawa 2001
- M. Ashby, D.R. Jones, „Materiały inżynierskie” Wydawnictwo Naukowo Techniczne, Warszawa 1996
- D.G. Morris “Mechanical Behaviour of nanostructured Materials” Trans Tech Publication, Zurich 2001
- A.Inoue, “Bulk Amorphous Alloys”, Trans Tech Publications, Zuerich, 1999
- H. Buhl, “Advanced Aerospace Materials”, Springer Verlag, Berlin, 1992
- H. Morawiec, Z. Lexton, “Materiały z pamięcią kształtu do zastosowań biomedycznych” Wydawnictwo Politechniki Śląskiej 2011
- J. Polmear, „Light Alloys” Amsterdam 2006

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## **Scientific Seminar**

(10 h, credit, 1 ECTS)

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## **Consultations with Supervisor**

(2 h, credit, 1 ECTS)



## Year 3

### Semester I

#### Course: **Chemical and kinetic characterization of diffusional phase transformations**

#### **(Charakterystyka chemiczna i kinetyczna dyfuzyjnych przemian fazowych)**

(15 h, exam, 2 ECTS)

#### *Course description*

1. Fundamentals of diffusion processes: Continuum Theory of Diffusion-Fick's Laws, Solutions of the Diffusion Equation.
2. Point Defects in Crystals: Pure Metals, Substitutional Binary Alloys, Intermetallics.
3. Diffusion Mechanisms: Interstitial Mechanism, Collective Mechanisms, Vacancy Mechanism, Divacancy Mechanism, Interstitialcy Mechanism, Interstitial-substitutional Exchange Mechanisms.
4. Diffusion in Metallic Materials: Self-diffusion in Metals, Diffusion of Interstitial Solutes in Metals, Interdiffusion (chemical diffusion) and Kirkendall Effect, Reactive diffusion.
5. Dependence of Diffusion on Temperature and Pressure.
6. Diffusion along High-Diffusivity Paths and in Nanomaterials.
7. Chemical and kinetic characterization of diffusional phase transformations: determination of interdiffusion coefficient, diffusion couple technique, diffusion soldering (growth kinetics) – theory and examples.

#### *Course is based on the following literature:*

- H. Mehrer, Diffusion in Solids Fundamentals, Methods, Materials, Diffusion-Controlled Processes, Springer-Verlag Berlin Heidelberg 2007, ISBN 978-3-540-71486-6.
- V.I. Dybkov, Reaction diffusion and solid state chemical kinetics, The IPMS Publications Kyiv 2002, ISBN 966-02-2545-8.

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## Course: **Thermo-mechanical processing of metallic materials**

### **(Procesy termomechaniczne materiałów metalicznych)**

(15 h, exam, 2 ECTS)

#### ***Course summary***

A series of Courses briefly recalls the basic description, definitions and elementary constitutive laws used to describe plastic deformation. Then it covers a description of work hardening at relatively low temperatures (where thermally activated processes do not play a key role) followed by the analysis of some important features of plastic deformation significant for large strains (**Course 1 & 2**).

Softening processes (recovery, recrystallization and grain growth) and associated microstructural changes will be discussed based on driving force and involved mechanisms. This part provides an overview of several essential parameters including: stored energy of deformation, surface energy and the movement of high-angle boundaries (**Course 3**).

**Course 4** will be dedicated to the description and interpretation of crystallographic textures. After an introduction to the 'world' of graphical representation of texture data, a short survey of the most important cold deformation and recrystallization textures will be presented.

**Course 5** will be dedicated to analysis of the mechanisms of band like strain inhomogeneities formation in fcc metals (deformation, transition and shear bands) and their influence on overall deformation and recrystallization textures in fcc metals will be thoroughly discussed. Particular attention will be paid to the description of the fundamental mechanisms responsible for cube texture formation during recrystallization of copper and aluminum base alloys.

**Course 6 (Technology)** In this part the most important metal forming operations - rolling, forging, extrusion, sheet metal forming and wire drawing will be described. The fundamental aspects of the processing will be discussed together with the details on the machines, die-tool design and process optimization. All of such issues are covered based on their relevance to the respective forming techniques.

**Course 7 (Case studies)**. In a number of examples the thermo-mechanical processing of some products will be analysed and discussed. These case studies show how properties can be successfully optimized by a carefully control of the microstructure during processing. Example from Al-industry include the processing of beverage cans, whereas thermo-mechanical

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processing of steel products will be illustrated with a case study of steel for car body (IF, dual phase and TRIP-steels).

### ***Course description***

1. Plasticity and work hardening
2. Instability of isotropic/anisotropic materials in tensile test and under biaxial stresses
3. Softening mechanism: recovery, recrystallization and grain growth
4. Texture development during thermo-mechanical processing. Deformation vs. recrystallization textures
5. Plastic flow instabilities formation
6. Technology: rolling, forging, extrusion, sheet metal forming and wire drawing.
7. Thermo-mechanical processing - example of aluminum and steel flat products processing.

### ***Course is based on the following literature:***

- D. Hull, Introduction to dislocations.
- A. Kelly, G.W. Groves, Crystallography and crystal defects.
- F.J. Humphreys, M. Hatherly, Recrystallization and related annealing phenomena.
- O. Engler, V. Randle, Introduction to texture analysis, macrotexture, microtexture and orientation mapping.
- P.F. Thomason, Ductile fracture of metals.
- W.A. Backofen, Deformation processing.

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### **Foreign language**

(30 h, credit, 1 ECTS)

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### **Scientific Seminar**

(10 h, credit, 1 ECTS)

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### **Consultations with Supervisor**

(2 h, credit, 1 ECTS)

## Semester II

Course: **Surface engineering – processing by directed energy techniques**

**(Inżynieria powierzchni - procesy generowane technikami o ukierunkowanej energii)**

(15 h, exam, 2 ECTS)

### *Course summary*

Multicomponent, nanostructured and functionally graded coatings or thin films may exhibit unique physical, mechanical, chemical properties ensuring remarkable degradation resistance where the surface protection of materials against wear, corrosion, friction is a key issue. A broad overview on modern coatings and thin-film deposition technique is presented. The major aim of these Courses is to show and discuss various problems of physics and chemistry involved in the production, characterization and applications of coatings and thin films, which can be variously hard and wear resistant. Attention is paid at the bio-medical coating for tissue contacting materials. A balance is found between fundamental aspects and experimental results illustrating various models, mechanisms and theories. New trends and new results are also evoked to have an overlook about future developments and applications.

### *Course description*

1. Scope of „surface engineering”
2. Modern methods of technological surface layers fabrication
3. Pressure units - Vacuum
4. Mechanical methods of surface modification
5. Chemical methods of surface modification CVD (chemical vapour deposition)
6. Solidification from the gaseous phase
7. Plasma
8. Physical methods of surface modification PVD (physical vapour deposition)
9. Ion-electron interaction with solid surface
10. Laser beam-solid surface interaction
11. Magnetron discharge in plasma processing
12. Surface modification by ion interaction

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13. Surface modification by plasma ion implantation
14. Surface modification by low-energy and high-current electron beam
15. Surface modification by laser remelting and alloying
16. Laser rapid prototyping
17. Pulsed laser deposition using laser ablation
18. Surface cleaning by laser ablation
19. Surface modification by thermal plasma
20. Arc evaporation
21. Methods of surface diagnostics
  - a. spectroscopic method - Raman spectroscopy
  - b. structural (AFM, SEM, TEM, confocal microscopy CLSM)
  - c. residual stress and methods of measurements
  - d. micro-mechanical properties
22. Hard and super hard coatings on the basis of: nitrides, carbides, borides and nano-composites
23. Surface thermal barriers
24. Polymer coatings fabricated by plasma polymerization
25. Trends in surface engineering in the world

***Course is based on the following literature:***

- M.Ashby: Materials; engineering, science, processing and design, Elsevier 2010
- Y.Pauleau: Materials Surface Processing by Directed Energy Techniques, Elsevier 2006
- T.Burakowski, T.Wierzchoń: Inżynieria powierzchni metali, Wyd.nauk.-Techn. 1995
- M.Blicharski: Wstęp do inżynierii materiałowej, Wyd. Nauk.-Techn. 2003
- L.A.Dobrzański: Metalowe materiały inżynierskie, Wyd. Nauk.-Techn., 2004
- Mazurkiewicz: Nanonauki i Nanotechnologie, Wyd.Inst.Technol.Ekspl., Radom 2007

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## Course: **Photovoltaic systems – theory and practice**

(Systemy PV – teoria i praktyka)

(15 h, exam, 1 ETCS)

### ***Course summary***

Renewable sources of energy are those whose use is not associated with the long-term deficit and their stock is renewed in a short time. Solar radiation, winds, geothermal, biomass, tides belong to renewable resources of energy called simply renewable energies.

The use of renewable energies can reduce greenhouse gas emissions and can ensure the security of energy supply. Therefore, the transformation of the old energy system based on fossil fuels to a fully renewable global energy system is a very urgent task. Among different renewable energy technologies, solar photovoltaics (PV) has a particularly promising future.

The course concerns in the field of materials engineering dedicated for photovoltaic, which at present is one of the most dynamically developed domains of science and industry. Despite of huge progress and many investigation results of solar cells belonged to 2nd and 3rd generation, still there is a big scientific interest in first generation photovoltaic structures what makes that they are the most popular in the mass production [EPIA REPORT 2013-2017]. Dominating role of this type of solar cells is a result of practical causes. One of them is that, the technology of the preparation of the 1st generation solar cells based on silicon wafer and screen printing method are already well known, what allow on manufacturing of photovoltaic structures with high efficiency of solar energy conversion. Topics of lectures concern the technology of solar energy and wind energy to produce electrical energy.

### ***Course description***

1. Introduction to photovoltaics (K. Drabczyk - 2h)

The lecture concerns basic information about the solar energy and photovoltaic energy conversion.

2. Technology of solar cells (P. Panek - 5 h)

In this lecture the industrial technology of silicon solar cells and thin films solar cells will be presented.

3. Photovoltaic systems K. Drabczyk - 3h)

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The lecture concerns the technology, applications, economics of photovoltaic systems and materials engineering dedicated for photovoltaic.

4. Emerging photovoltaics (M. Lipiński - 5 h).

The lecture concerns emerging materials and devices including dye-sensitized solar cell, organic solar cell, perovskite solar cell and quantum dot solar cell.

***Course is based on the following literature:***

- Luque and S. Hegedus (Editors), Handbook of Photovoltaic Science and Engineering, (2003 John Wiley & Sons).
- Kazimierz Drabczyk, Piotr Panek, Silicon-based solar cells. Characteristics and production processes (2012 Kraków, IMIM ).
- L. Tsakalagos (Editor), Nanotechnology for Photovoltaics (2010) Taylor & Francis Group, LLC, New York).
- A. Marti, A. Luque (Editors), Next Generation Photovoltaics (2004) Institute of Publishing Bristol and Philadelphia).
- Junfen Yan and Brian R. Saunder, Third-generation solar cells: a review and comparison of polymer:fullerene, hybrid polymer and perovskite solar cells, RSC Adv., (2014), 4, 43286.
- Peng Gao, Michael Gratzel and Mohammad K. Nazeeruddin, Organohalide lead perovskites for photovoltaic applications, Energy Environ. Sci., (2014), 7, 2448.

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### **Foreign language**

(30 h, credit, 1 ECTS)

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### **Scientific Seminar**

(10 h, credit, 1 ECTS)

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### **Consultations with Supervisor**

(2 h, credit, 1 ECTS)



## Year 4

### Semester I

Course: **Structural effects of phase transformations**

**(Efekty strukturalne przemian fazowych)**

(15 h, exam, 1 ECTS)

#### *Course description*

##### **1. Principles of solidification**

Homogeneous nucleation; heterogeneous nucleation; nucleation and growth in solid-state reactions

##### **2. Transformations in solids**

Description of overall transformation; time-temperature-transformation diagrams

##### **3. Transformation to stable phases**

The Fe-Fe<sub>3</sub>C phase diagram; isothermal transformations in steels

##### **4. Transformation to stable phases**

The eutectoid reaction; phases and composition of pearlite; hypo- and hypereutectoid steels; spinodal decomposition

##### **5. Transformation to transient phases**

Controlling the eutectoid reaction; the bainitic reaction; the martensitic reaction and tempering

#### *Course is based on the following literature:*

- W.D. Callister Jr, D.R. Rethwisch, Materials Science and Engineering, Ninth Edition, John Wiley, 2015
- A.G.Guy Introduction to materials science, McGraw-Hill 1972
- Donald R.Askeland, Pradeep P. Phule, The science and engineering of materials, Thomson Canada Limited 2006

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Course: **Orientation characteristics of materials microstructure**  
(**Orientacja krystalograficzna w badaniach mikrostruktury**)

(15 h, exam, 1 ECTS)

*Course description*

**1. Orientation characteristics of the microstructure of materials - TEM.**

Repetition of basic concepts of Orientation Imaging Microscopy: crystallographic orientation, and texture; misorientation, and grain boundary characterization; Image Quality Factor, and microstrains.

**2. Formation of the diffraction pattern in the TEM.**

Selected area (electron) diffraction *SAED*, Convergent Beam Electron Diffraction *CBED*, Large-angle Convergent Beam Electron Diffraction (Kossel patterns) *LACBED*, Kikuchi patterns.

**3. Orientation Imaging Microscopy in TEM.**

An interactive software for simulation, indexing and analysis of various types of TEM diffraction patterns - [crystorient.com](http://crystorient.com).

**4. Second and third order stresses in TEM and SEM.**

A critical analysis of stress measurement by *CBED/TEM*, effect of elastic strain gradient on Kikuchi line width, different types of image quality factors, as a tool for estimating third order stresses.

**5. Orientation Imaging Microscopy in practice. Part I.**

Example I: *In situ* TEM observation of the recrystallization in fcc alloys with low stacking fault energies.

Example II: Recrystallization of aluminium alloy with bimodal second-phase particle distribution.

**6. Orientation Imaging Microscopy in practice. Part II.**

Example III: Microstructure and texture effects generated during plastic deformation by complex loading of titanium. Example IV: New approach to misorientation distribution function analysis - the strength of interfaces as a key for rational composite design.



***Course is based on the following literature:***

- Morawiec A. (2004): *Orientations and Rotations. Computations in Crystallographic Textures*. Berlin, Heidelberg, New York: Springer-Verlag.
- Williams D.B., Barry Carter C. (2004): *Transmission Electron Microscopy; Basics*, Springer.
- Morniroli J-P. (2002) *Large-angle convergent-beam diffraction(LACBED)*, Société Française des Microscopies, Paris.
- K. Sztwiertnia (2009) *Orientacja krystalograficzna w badaniach mikrostruktury materiałów*, Polska Akademia Nauk, Instytut Metalurgii i Inżynierii Materiałowej im. Aleksandra Krupkowskiego, Kraków 2009, ISBN 978-83-607 68-02-0.

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**Course: Introduction to economy - selected issues**

(30 hours, exam, 1 ECTS)

The final topics and a short description will be given in due course.

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**Scientific Seminar**

(10 h, credit, 1 ECTS)

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**Consultations with Supervisor**

(2 h, credit, 1 ECTS)

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**Semester II**

**Scientific Seminar**

(10 h, credit, 1 ECTS)

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**Consultations with Supervisor**

(2 h, credit, 1 ECTS)





**PhD procedure will be launched at the latest after completing Semester II of the Year 3 of the Studies. The procedure begins with the PhD seminar during which the PhD student is obliged to present a substantial progress of the PhD thesis.**

**The PhD thesis is to be written in English.**