



Corso di Laurea in
**SCIENZA E TECNOLOGIA
 DEI MATERIALI**

Triennale – L30

General information	
Academic subject	X-ray powder Diffraction
Degree course	<i>Material's science and technologies</i>
Academic Year	2021-2022
European Credit Transfer and Accumulation System (ECTS)	4
Language	<i>Italian</i>
Academic calendar (starting and ending date)	<i>2nd semester, 2nd year</i>
Attendance	<i>not required</i>

Professor/ Lecturer	
Name and Surname	Ernesto Mesto
E-mail	ernesto.mesto@uniba.it
Telephone	+390805442597
Department and address	<i>Department of Earth Sciences and Geoenvironmental</i>
Virtual headquarters	http://www.geo.uniba.it/mesto.html
Tutoring (time and day)	Office hours, by appointment via email

Syllabus	
Learning Objectives	<i>Acquiring fundamental knowledge about the X-ray Powder diffraction by polycrystalline materials. Carry out an X-ray data collection from a powder and derive structural information about the sample.</i>
Course prerequisites	<i>Detailed knowledge of single crystal X-ray diffraction.</i>
Contents	<p>Fundamentals of diffraction <i>Recalls on the crystals, symmetry and reciprocal lattice. Bragg's law. Ewald sphere. Origin of a powder diffraction pattern. Structural information from a diffractogram.</i></p> <p>Experimental techniques <i>Automatic X-ray diffractometers for powder analyses. Bragg-Brentano geometry. Reflection and transmission scans. X-ray sources: X-ray tube and synchrotron light. Sources of neutrons. Monochromatizing of X-rays. Collimators, Soller Slits, divergent and antiscattering slits. Point, linear and areal X-ray detectors. Sample preparation. Instrumental errors. Data collection strategy.</i></p> <p>Interpretation of a powder diffraction pattern <i>Data reduction: background subtraction, smoothing, $K\alpha_2$ stripping. Peak search methods. Identification of crystal phases: Hanawalt and automatic method. Crystallographic databases. Indexing of a powder diffraction pattern. Determination of the unit cell. Determination of the space group.</i></p> <p>Quantitative analysis of polyphasic mixtures <i>Intensity of Bragg Peak. Structure factor. Standard addition method. Internal standard method. RIR method (Reference Intensity Ratio). Rietveld refinement. Quantification of the amorphous component. Indirect and direct method. Single</i></p>



Corso di Laurea in **SCIENZA E TECNOLOGIA DEI MATERIALI**

Triennale – L30

	<p><i>peak method. Internal Standard Method. External standard method. PONKCS method. Degree of crystallinity.</i></p> <p>Application of X diffraction <i>XRD characterization of materials of industrial interest: the case of clinker of Portland cement. Guided experimental measurements in the laboratory: data collection of polyphasic samples, phase identification and quantitative analysis with the RIR and Rietveld method.</i></p>
Books and bibliography	<i>V. K. Pecharsky and P.Z. Zavaliy. Fundamentals of powder diffraction and structural characterization of materials, 2nd Edition, Springer, New York, 2009.</i>
Additional materials	

Work schedule			
Total	Lectures	Hands on (Laboratory, working groups, seminars, field trips)	Out-of-class study hours/ Self-study hours
Hours			
100	24	15	61
ECTS			
4	3	1	
Teaching strategy		<i>Lectures and laboratory experiences.</i>	
Expected learning outcomes			
Knowledge and understanding on:		Definition of polycrystalline material. Fundamental aspects of X-ray powder. Knowledge of the descriptive parameters of a diffractogram and of the information that can be derived from it.	
Applying knowledge and understanding on:		X-ray data collection of powder samples. Elaboration of diffraction patterns of powder samples.	
Soft skills		<ul style="list-style-type: none"> • <i>Making informed judgments and choices</i> Critic analysis of a diffraction powder pattern. • <i>Communicating knowledge and understanding</i> Presentation and dissemination of the crystallographic knowledge with an appropriate scientific language. Presentation of the results obtained from the interpretation of a powder diffraction pattern; ability to integrate quickly and effectively in the workplace and in scientific research laboratories. • <i>Capacities to continue learning</i> Learning and transfer of experimental x-ray powder diffraction protocol and of data analysis of powder diffraction patterns. 	

Assessment and feedback	
Methods of assessment	<i>Oral examination (100%)</i>
Evaluation criteria	<ul style="list-style-type: none"> • <i>Knowledge and understanding</i> <ul style="list-style-type: none"> ○ Minimum level: Knowledge about the origin of a powder diffraction pattern, Bragg's Law, Phase identification. Hanawalt method. ○ Intermediate level: Qualitative analysis of a powder diffraction pattern: Search and Match algorithms. Quantitative analysis of a powder diffraction pattern of a polyphasic mixture. Knowledge of the experimental apparatus used to collect the X diffraction pattern of a powder material. Data acquisition.



Corso di Laurea in
**SCIENZA E TECNOLOGIA
 DEI MATERIALI**

Triennale – L30

	<ul style="list-style-type: none"> ○ Upper level: Space group determination. Solution of the phase problem using powder diffraction data. Rietveld method: structural determination of a powder sample. Quantitative analysis by Rietveld analysis. Determination of the amorphous content. ● <i>Autonomy of judgment</i> <ul style="list-style-type: none"> ○ For the intermediate level: Qualitative and quantitative analysis of a powder diffraction. ○ For the upper level: Structural determination with powder diffraction data. ● <i>Communicating knowledge and understanding</i> <ul style="list-style-type: none"> ○ For all levels: The student should properly discuss about the x-ray powder diffraction in an oral examination. Topics with an increasing degree of depth will be proposed in order to establish the student's level of knowledge. ● <i>Communication skills</i> <ul style="list-style-type: none"> ○ use of correct scientific terminology. Language skills required to discuss the topics of the course.
Criteria for assessment and attribution of the final mark	<p><i>Reaching the minimum level will result in a final grade between 18-20.</i> <i>Reaching the intermediate level will result in a final grade between 21-26.</i> <i>Reaching the higher level will result in a final grade between 27-30.</i></p>
Additional information	