GUJARAT TECHNOLOGICAL UNIVERSITY (GTU)

Competency-focused Outcome-based Green Curriculum-2021 (COGC-2021)
Semester-V

Course Title: Material Characterization
(Course Code: 4352107)

Diploma Programme in which this course is offered	Semester in which offered
Metallurgy Engineering	5th Semester

1 RATIONALE

Metallurgy engineers must possess a deep understanding of composition-structure-property relationship of materials. Therefore, it becomes essential for them to be familiar with various techniques to analyze the composition, properties as well as structure from atomic to macroscopic level. Material characterization includes study of techniques such as optical and electron microscopy, X-ray diffraction, thermal analysis, and mechanical testing. The subject will enable the students to get information on composition, crystal structure, defects, morphology, volume and orientation of phases, phase transformation, residual stress etc. This information can then be related to the physical, thermal and mechanical behavior of materials. Such knowledge is critical for selecting materials, developing new materials, improving existing ones in order to meet specific performance requirements as per the standards and specifications for various applications. Material characterization is also an essential aspect for quality control and troubleshooting problems during manufacturing. Since material characterization is a broad field, the present curriculum is designed to cater diploma engineering students.

2 COMPETENCY

The course content should be taught and curriculum should be implemented with the aim to develop required skills in students so that they are able to acquire following competencies.

- **Implement suitable technique for qualitative and quantitative analysis of composition, structure and properties of materials for material selection and quality control purposes.**

3 COURSE OUTCOMES (COs)

The theory should be taught and practical should be performed in such a manner that students are able to acquire different learning outcomes in cognitive, psychomotor and affective domain to demonstrate following course outcomes:

CO1: Classify various methods of material characterization
CO2: Use optical and scanning electron microscopy (SEM) for qualitative and quantitative analysis of microstructure
CO3: Understand the use of X-ray diffraction (XRD) technique for materials characterization
CO4: Determine the chemical composition of materials using Energy Dispersive Spectroscopy (EDS), Spark Emission Spectroscopy and X-ray Fluorescence (XRF)

4 TEACHING AND EXAMINATION SCHEME

Teaching Scheme (In Hours)			Total Credits (L+T+P/2)	Examination Scheme				
				Theory Marks		Practical Marks		Total Marks
L	T	P	C	CA	ESE	CA	ESE	
2	0	2	3	30*	70	25	25	150

(*): Out of 30 marks under the theory CA, 10 marks are for assessment of the micro-project to facilitate integration of COs and the remaining 20 marks is the average of 2 tests to be taken during the semester for the assessing the attainment of the cognitive domain UOs required for the attainment of the COs.

***Legends:** **L**-Lecture; **T** – Tutorial/Teacher Guided Theory Practice; **P** -Practical; **C** – Credit, **CA** - Continuous Assessment; **ESE** -End Semester Examination.*

5 SUGGESTED PRACTICAL EXERCISES

Sr. No.	Practical Outcomes (PrOs)	Unit No.	Approx. Hrs. Required
1	Prepare a metallographic specimen and examine its microstructure using optical microscope	II	4
2	Observe a metallographic specimen under bright field, dark field and polarized light microscopy	II	4
3	Measure the grain size, volume of phases, coating thickness measurement and decarburization depth in a metallic specimen using image analyzer as per ASTM E 1382, ASTM E 1245, ASTM B 487 and ASTM E 1077 standards	II	4
4	Observe a metallic specimen using scanning electron microscope	III	4
5	Demonstrate the process of phase identification in metallic sample from its X-ray diffraction pattern	IV	4
6	Determine the chemical composition of a metallic sample using SEM-EDS as per ASTM E 1508 standard	V	4
7	Determine the chemical composition of a metallic sample using Positive Material Identification (PMI) (X-Ray Fluorescence) method and spark emission spectrometer as per ASTM E 1476 standard	V	4
	Total Hours		**28**

Notes:

1. *More Practical Exercises can be designed and offered by the respective course teacher to develop the industry relevant skills/outcomes to match the COs. The above table is only a suggestive list.*
2. *The following are some sample 'Process' and 'Product' related skills (more may be added/deleted depending on the course) that occur in the above listed Practical Exercises of this course required which are embedded in the COs and ultimately the competency.*

Sr. No.	Sample Performance Indicators for the PrOs.	Weightage in %
1	Identification of the component and understand test procedure/experimental set-up	20
2	Operate equipment set-up	10
3	Observation and recording of the data correctly	10
4	Interpretation of the result and conclusion	20
5	Safety precaution and safety gadgets used	20
6	Submission of report within time limit and attendance in the laboratory	20

6 MAJOR EQUIPMENT/ INSTRUMENTS REQUIRED

These major equipment with broad specifications for the PrOs is a guide to procure them by the administrators to user in uniformity of practical's in all institutions across the state.

S. No.	Equipment Name with Broad Specifications	PrO. No.
1	Metallurgical microscope Magnification: 50X to 1000X Objective Lenses: 5x, 10x, 20x, 50x and 100x magnification Eyepiece: 10x magnification Calibration Standards (Calibration scale) Image Analyzer with camera: To capture and analyze digital images. The software should allow for measuring and analyzing features such as grain size, phase identification and length measurement. The microscope should have the capability to use a range of contrast methods, including bright field, dark field, and polarized light.	1, 2 and 3
2	Scanning Electron Microscope (SEM) with EDS probe: Magnification range: up to 2,00,000 X Accelerating voltage range: 0.1 to 30 kV Imaging modes: SE, BSE, EDS	4, 6
3	X-ray Diffraction (XRD) instrument: Operating range: 2θ = 0 to 130 degrees	5

S. No.	Equipment Name with Broad Specifications	PrO. No.
4	PMI (Positive Material Identification) machine that uses the XRF (X-Ray Fluorescence) method A standard calibration piece shall be available that have a known composition to ensure accurate and reliable results. Safety features to protect the operator from exposure to X-rays.	7
5	Spark Emission Spectrometer: Channels: For Ferrous and non-ferrous metals and alloys Sample holder: Solid samples Detector type: Photomultiplier tube or CCD	7

7 AFFECTIVE DOMAIN OUTCOMES

The following sample Affective Domain Outcomes (ADOs) are embedded in many of the above-mentioned COs. More could be added to fulfill the development of this course competency

a) Develop an interest in material characterization and appreciate its importance in various fields of science and engineering.
b) Follow standard methods for calibration and testing to ensure accuracy and precision in test results.
c) Follow safety protocols when handling various materials and instruments, especially when working with harmful chemicals and radiation.
d) Develop critical thinking and problem-solving skills to analyze and interpret data from material characterization techniques and make informed decisions.
e) Develop ethical awareness and understand the ethical implications of material characterization techniques and their applications in various fields.

8 UNDERPINNING THEORY

The major underpinning theory is given below based on the higher level UOs of Revised Bloom's taxonomy that are formulated for development of the COs and competency. If required, more such UOs could be included by the course teacher to focus on attainment of COs and competency.

Unit	Major Learning Outcomes	Topics and Sub-topics
UNIT-I Introduction to material characterization	1a. Describe the importance of material characterization 1b. Classify various techniques of material characterization	1.1. Introduction and importance of material characterization 1.2. Classification of material characterization techniques
UNIT-II Optical microscopy	2a. Explain the principle, construction and working of metallurgical microscope 2b. Distinguish between bright	2.1. Principle, construction and working of metallurgical microscope 2.2. Bright field and dark field

	field and dark field illumination 2c. Calculate the magnification, resolution, depth of focus and Numerical aperture of an optical microscope 2d. Application of polarized light and colour metallography	illumination 2.3. Magnification, resolution and depth of focus and Numerical aperture 2.4. Polarized light microscopy 2.5. Colour metallography
UNIT-III Scanning Electron Microscopy	3a. Describe the principle, construction and working of scanning electron microscope 3b. Explain the Interactions between electron beam and sample in SEM 3c. State the applications and limitations of SEM	3.1. Principle, construction and working of scanning electron microscope (SEM) 3.2. Interactions between electron beam and sample in SEM 3.3. Applications and limitations of SEM
UNIT-IV X-ray Diffraction	4a. Explain the production of X-rays and the characteristics of X-ray spectra. 4b. Explain the principles of absorption and diffraction of X-ray by matter 4c. Describe XRD methods 4d. Use of XRD method in material characterization	4.1. X-ray generation, continuous and characteristic spectrum 4.2. Absorption of X-ray by matter and diffraction of X-ray – Bragg's law 4.3. X-ray diffraction (XRD) methods 4.4. Application of X-ray diffraction (XRD) methods
UNIT-V Chemical composition analysis	5a. Determine the chemical composition of materials using X-ray energy dispersive spectroscopy (EDS) 5b. State the applications and limitations of EDS 5c. Determine the chemical composition using spark emission spectroscopy 5d. State the applications and limitations of spark emission spectroscopy 5e. Understand use of Positive Material Identification (PMI)	5.1. Principle and working of energy dispersive spectroscopy (EDS) 5.2. Applications and limitations of EDS 5.3. Principle and working of spark emission spectroscopy applications and limitations of spark emission spectroscopy 5.4. Application of Positive Material Identification (PMI) using XRF (X-ray fluorescence)

9 SUGGESTED SPECIFICATION TABLE FOR QUESTION PAPER DESIGN

Unit	Unit Title	Teaching Hours	Distribution of Theory Marks			
			R Level	U Level	A Level	Total Marks
I	Introduction to material characterization	02	3	4	0	07
II	Optical microscopy	08	7	7	6	20
III	Scanning Electron Microscopy	06	6	7	3	16
IV	X-ray Diffraction	06	5	7	4	16
V	Chemical composition analysis	06	4	4	3	11
Total		**28**	**25**	**29**	**16**	**70**

Legends: R = Remember; U = Understand; A = Apply and above levels (Bloom's revised taxonomy)
Notes:

1. This specification table shall be treated as a general guideline for students and Teachers. The actual distribution of marks in the question paper may slightly vary from above Table.
2. Ask the questions from each topic as per marks weightage. Numerical questions are to be asked only if it is specified. Optional questions must be asked from the same topic.

10 SUGGESTED STUDENT ACTIVITIES

Other than the classroom and laboratory learning, following are the suggested student-related co-curricular activities which can be undertaken to accelerate the attainment of the various outcomes in this course: Students should conduct following activities in group

1. Visit a metallurgical testing lab to observe the calibration and testing techniques of various material characterization equipment.
2. List out sample preparation requirements for various material characterization techniques.
3. Identify the suitable technique/s for quantitative and qualitative analysis of various microstructural features of a metallic material.
4. Prepare a list of relevant ASTM standards for microstructural analysis of metallic specimens using optical and scanning electron microscopy.
5. List out the limitations of EDS and spark emission spectrometer for compositional analysis.
6. Group discussion on recent developments in existing techniques of material characterization.

11 SUGGESTED SPECIAL INSTRUCTIONAL STRATEGIES (if any)

These are sample strategies, which the teacher can use to accelerate the attainment of the various outcomes in this course:

a) Massive open online courses (MOOCs) may be used to teach various topics/sub topics.
b) Guide student(s) in undertaking micro-projects
c) 'L' in section No. 4 means different types of teaching methods that are to be employed by teachers to develop the outcomes.
d) About 20% of the topics/sub-topics which are relatively simpler or descriptive in nature is to be given to the students for self-learning, but to be assessed using different assessment methods.
e) With respect to section No.10, teachers need to ensure to create opportunities and provisions for co-curricular activities.
f) Guide students on how to address issues on environment and sustainability.
g) Encourage students to read codes and standards.

12 SUGGESTED MICRO-PROJECTS

Only one micro-project is planned to be undertaken by a student that needs to be assigned to him/her in the beginning of the semester. In the first four semesters, the micro-project are group-based. However, in the fifth and sixth semesters, it should be preferably be individually undertaken to build up the skill and confidence in every student to become problem solver so that he/she contributes to the projects of the industry. In special situations where groups have to be formed for micro-projects, the number of students in the group should not exceed three.

The micro-project could be industry application based, internet-based, workshop-based, laboratory-based or field-based. Each micro-project should encompass two or more COs which are in fact, an integration of PrOs, UOs and ADOs. Each student will have to maintain dated work diary consisting of individual contribution in the project work and give a seminar presentation of it before submission. The total duration of the micro-project should not be less than 16 (sixteen) student engagement hours during the course. The student ought to submit micro-project by the end of the semester to develop the industry-oriented COs.

A suggestive list of micro-projects is given here. This has to match the competency and the COs. Similar micro-projects could be added by the concerned course teacher:

1. A literature review of various material characterization techniques including their advantages and limitations.
2. Analysis of microstructure and grain size of various alloys using image analysis software on pre-existing images available in databases or online resources.
3. Prepare a report on the information obtained from each type of interaction between electron beam and metallic sample in SEM.
4. Prepare a report on XRD testing of a powder and solid sample.
5. Compare EDS and spark emission spectrometry techniques for compositional analysis of a metallic specimen.

13 SUGGESTED LEARNING RESOURCES

Sr. No.	Title of Book	Author	Publication with place, year and ISBN
1	An Introduction to Material Characterization	P. R. Khangaonkar	Publisher: Penram International Publishing (India) Pvt. Ltd. Year: 2008 ISBN-10: 8187972807
2	A Guide to Materials	John P. Sibilia	Publisher: Wiley-VCH

	Characterization and Chemical Analysis		Year: 1996 ISBN-13: 0471186333
3	Elements of X-Ray Diffraction	B.D. Cullity	Publisher: Addison – Wesley Publishing Company Inc. Year: 2001 ISBN-10: 0201011743
4	Materials Characterization: Introduction to Microscopic and Spectroscopic Methods	Yang Lang	Publisher: Wiley-VCH Year: 2013 ISBN-13: 9783527334636
5	ASM Handbook, Volume 10: Materials Characterization	ASM International	Publisher: ASM International Year: 2019 ISBN-13: 9781627082112

14 SOFTWARE/LEARNING WEBSITES

- https://www.doitpoms.ac.uk/miclib/index.php
- https://onlinecourses.nptel.ac.in/noc22_mm14/preview
- https://archive.nptel.ac.in/courses/112/106/112106227/
- https://archive.nptel.ac.in/courses/113/106/113106064/
- https://emb-iitk.vlabs.ac.in/exp/sem-basics/theory.html
- https://atelearning.com/XRLab/presentation_3/index.htm

15 PO-COMPETENCY-CO MAPPING

Semester V	Material Characterization (Code: 4352107)						
	POs						
Competency & Course Outcomes	**PO 1 Basic & Discipline specific knowledge**	**PO 2 Problem Analysis**	**PO 3 Design/ develop-ment of solutions**	**PO 4 Engineering Tools, Experimentation & Testing**	**PO 5 Engineering practices for society, sustainability & environment**	**PO 6 Project Manage-ment**	**PO 7 Life-long learning**
Competency	Implement suitable technique for qualitative and quantitative analysis of composition, structure, and properties of materials for material selection and quality control purposes						
CO1: Classify various methods of material characterization	3	-	-	-	-	-	1
CO2: Use optical and scanning electron microscopy (SEM) for qualitative and quantitative analysis of microstructure	3	2	2	3	1	-	1

CO3: Understand the use of X-ray diffraction (XRD) technique for materials characterization	3	2	2	3	1	-	1
CO4: Determine the chemical composition of materials using Energy Dispersive Spectroscopy (EDS), Spark Emission Spectroscopy and X-ray Fluorescence (XRF)	3	2	2	3	1	-	1

16 COURSE CURRICULUM DEVELOPMENT COMMITTEE

GTU Resource Persons

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