Digital Case Database Construction and Case Teaching for the Course of Modern Testing Technology of Materials

De Fang [1, 2, a], Jiayuan Wang [2, b], Tao Jing [2, c], Tianyi Gao [2, d], Caihong Lv [3, 4, e] *, and Han Zhou [1, f]

[1]Center for Materials Research and Analysis, Wuhan University of Technology, PR China;

[2] School of Materials Science and Engineering, Wuhan University of Technology, PR China;

[3] School of Printing & Packaging, Wuhan University, PR China;

[4] Jun An Forensic Center of Hubei, PR China.

[a] fangde0914@whut.edu.cn, [b] 336117@whut.edu.cn, [c] 335934@whut.edu.cn, [d] 335876@whut.edu.cn, [e] lvchwhu@163.com, [f] zhouhan2021@whut.edu.cn

Abstract. The course of modern testing technology of materials has the properties of strong comprehensive nature and practical characteristic. Case database construction and case teaching play significant roles in enhancing the teaching quality and effects of the course for teachers and students. Based on the methods and techniques for measuring materials, this course designs basic theoretical cases, enterprise production cases, laboratory ability verification project cases and cutting-edge cases of joint use technology, and introduces them into the classroom for implementation, and the related cases are collected to build the digital case database. On the one hand, based on the digital case database, the implementation of case teaching of this course overcomes the difficulty that the teaching content is relatively independent and the students are difficult to compare and analyze the similar methods and techniques for measuring materials, making the students' theoretical foundation more solid. On the other hand, based on the digital case database, the case teaching makes a comprehensive analysis of practical problems in production practice, stimulates students' creativity and interest, and encourages students to provide solutions for the development or improvement of testing means, thus improving the quality of courses and the effectiveness of education. According to the survey, more students were satisfied with case teaching for the course of modern testing technology of materials and their test scores had been significantly improved. Furthermore, the digital case database broaden the channels and ways for students to study the course, and help students realize the new learning mode of learning at any time, flexible learning and long-term learning.

Keywords: Case Database Construction; Case teaching; Testing technology; Creativity; Interest.

1. Introduction

The School of Materials Science and Engineering was founded in 1958 in Wuhan University of Technology. And the discipline of material science was listed as one of the key construction disciplines of the national Project 211 in 1996, one of the first national key disciplines in 2007, and one of the first national double first-class construction disciplines in 2016. It ranks A+ in the fourth round of national discipline assessment, and enters the top 1‰ of the world ESI discipline ranking. The School of Materials Science and Engineering of Wuhan University of Technology is one of the important bases for personnel training and scientific research of materials science and engineering in China.

The course of modern testing technology of materials is a compulsory course for graduate students majoring in materials in most universities. Wuhan University of Technology is one of the first university to offer the referred course for graduate students. At present, this course has been offered for more than 30 years, playing the crucial role in the recruitment, training and employment of the graduate students (academic master, professional master, academic doctor). This course mainly covers the basic principles, test methods and experimental data processing methods of modern analytical techniques such as X-ray diffraction analysis (XRD) technology, electron microscopic analysis technology, surface analysis technology, vibration spectrum analysis technology and thermal analysis technology. The major and difficult points of the course are application of XRD software, principle of electron microscopic analysis, calibration of electron diffraction pattern index, principle and analysis and application of vibration spectrum analysis, analysis and application of photoelectron spectroscopy, differential scanning calorimetric analysis and application of material thermal analysis technology. However, this course involves more basic principles and the course content is relatively boring. In foreign countries, teachers engaged in scientific research use scientific instruments and experimental techniques related to scientific research results to teach, and take the initiative to introduce numerous teaching cases, thus deepening students' understanding of this course. In China, due to the lack of practical experience in instrument operation and enterprise production, this course is mainly taught by classroom theory explanation, and there are few cases in the principle and application of instruments. This course needs to be updated as new testing techniques are emerging all the time. The bridge between theory and practice is built by the case teaching to advocate the coordination of teaching theory and teaching practice. The characteristics of the course of modern testing technology of materials help students to reach the other side of understanding theoretical knowledge through such a bridge [1]. The case is a practical problem in the teaching situation, and different students will have different interpretations of the practical problem, and the answer to the practical problem is not unique. Therefore, the case is very suitable for communication and discussion, and can become an effective carrier for students to deepen relevant theories of student testing technology [2-4]. In order to promote students' understanding and learning of this course, this course will independently design basic theoretical cases, collect enterprise production cases, refine laboratory ability verification project cases, sort out cutting-edge cases of combined technology, and implement them in class. Furthermore, the related cases are collected to build the digital case database, providing the digital educational information resources for students.

2. Specific Research Objects and Contents

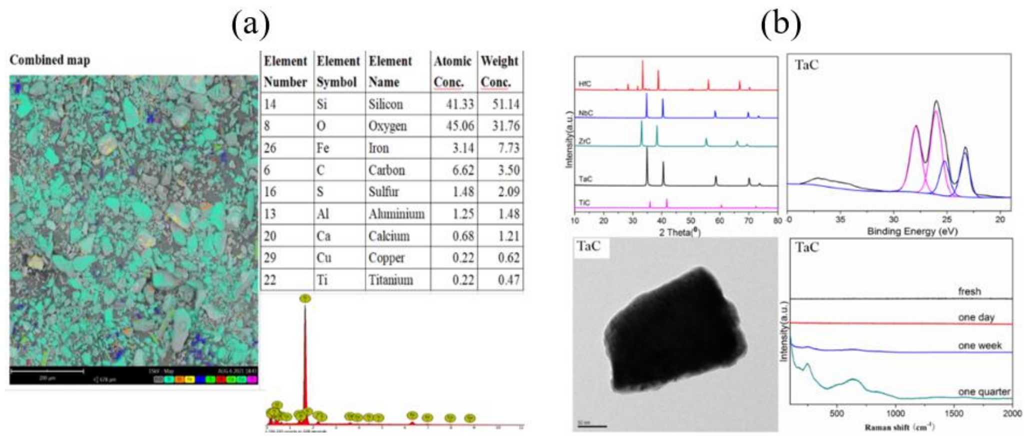
This study will be based on X-ray diffraction analysis technology, electron microscopic analysis technology, surface analysis technology, vibration spectrum analysis technology and thermal analysis and other modern technology content of the case design, collection, sorting and other work. Based on the basic principle and practical application of each test method in the syllabus, a basic theoretical case is designed. Based on the content of each chapter, we design, synthesize or collect a specific case independently, and we attach boring and profound theoretical knowledge to the case, so that students can systematically and deeply understand the corresponding test principles. The analysis difficulty of these cases is relatively small, and the answers have certain uniqueness. With the help of the related cases, students can learn and understand the analytical principles of XRD analyzer, scanning electron microscope (SEM), transmission electron microscope (TEM), electron probe X-ray microanalyzer (EPMA), X-ray photoelectron spectrometer (XPS), infrared spectrometer (IR), Raman spectrometer (Raman), thermogravimetric analyzer (TA), differential thermal analyzer (DTA), differential scanning calorimeter (DSC), X-ray fluorescence spectrometer (XRF) and other equipment. A comprehensive case is formed by comprehensively mining the theoretical knowledge and key testing techniques of enterprise production cases and university comparison project cases. The teaching content of each kind of testing equipment is relatively independent, and each kind of testing method has its unique principle and application. The actual production problems encountered by enterprises are generally more complex, and it must require a variety of testing methods to solve them together. Therefore, by collecting the production problems of enterprises and organizing them into teaching cases, the problems of relatively independent teaching content and difficult comparative analysis for students can be overcome. Moreover, the Center for Materials Research and Analysis is the China Inspection Body and Laboratory Mandatory Approval (CMA) unit, which needs to participate in some university review groups or the national certification and accreditation administration's inter-laboratory comparison plan every year.

This project will collect such resources to sort out high-quality cases, significantly improving the classroom effect and education effect. Combined with the new demand of new engineering teaching and the new trend of material research, the cutting-edge cases involving combined technology are collected. The types of testing technology in the existing teaching content can no longer meet the needs of the development of social production practice, and many of the latest testing equipment realize the combination of a variety of functional accessories, such as thermal analysis and mass spectrometry, thermal analysis and infrared, photoelectron spectroscopy and Raman spectroscopy, etc. In view of these latest co-use technologies, cutting-edge cases are made so that students can understand the latest co-use technology development and testing requirements. The construction of the digital case database will further broaden the channels and ways to study the course, and help students realize the new learning mode of learning at any time, flexible learning and long-term learning.

3. Results and Discussion

3.1 Basic Theoretical Case

Basic theoretical case is designed with the mixture of industrial raw materials. This case is mainly concerned with component analysis, and according to the various testing techniques covered in this course, EDS (Energy Dispersive X-Ray Spectrometer), XRF and XPS can quickly detect compositions of samples. The basic principle of EDS is that the characteristic X-rays emitted by different elements have different frequencies, suggesting that they have different energies. Therefore, the energy of different photons is detected and the elements can be determined (qualitative analysis). Based on the Moseley's law, each element has its characteristic X-rays of a specific wavelength (or energy). By measuring the wavelength (or energy) of the characteristic X-rays in the sample, it is possible to determine what elements are present in the sample, which is qualitative X-ray fluorescence spectrometer. The basic principle of XPS is below. After X-ray irradiation on the surface of the sample, photoelectrons are generated, and the chemical properties and composition of the sample surface are identified by determining the binding energy of the electrons. Although all three test methods can be used for composition analysis, their sample preparation and test costs differ greatly. EDS test is the fastest, and the cost is relatively low. XRF and XPS test cost is high, and they need to prepare a certain size of the sample. At the same time, XPS is the surface composition analysis and its analysis depth is less than 10 nanometers, while EDS and XRF are the bulk phase composition analysis and their test analysis depth is about 1 micron.



Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
14	Si	Silicon	41.33	51.14
8	O	Oxygen	45.06	31.76
26	Fe	Iron	3.14	7.73
6	C	Carbon	6.62	3.50
16	S	Sulfur	1.48	2.09
13	Al	Aluminium	1.25	1.48
20	Ca	Calcium	0.68	1.21
29	Cu	Copper	0.22	0.62
22	Ti	Titanium	0.22	0.47

Fig. 1 (a) EDS results of samples and (b) surface oxidation behavior of carbide metals.

In this case, the SEM/EDS was used for component analysis, and the resultant analysis information of the sample was obtained (as shown in Figure 1a), thus solving this technical requirement. Of course, if you choose XRF and XPS, you can also solve this problem, so the answer in this case is not unique. Through this basic theoretical case, students can understand the concept of component analysis and related testing principles, so they can choose suitable relevant testing methods. In the actual classroom teaching, most students also choose EDS test, and a few students choose XRF and XPS test, indicating that the students have a good grasp of the basic principles of component analysis.

3.2 Enterprise Production Cases

Enterprise production cases are from production reality, so the cases will have a coherent background presentation and overview of the entire problem. Therefore, the background of classic enterprise production case used in the class is shown below. A company purchased a batch of carbide metal as raw material. For some special reason, these raw materials had been shelved for half a year. In the process of production, the effect of this batch of raw materials become worse, and it was necessary to analyze the reason.

This is a practical production problem that involves a variety of information about the sample, such as phase, microstructure, and composition changes. Therefore, we put this case in the last few classes of this course to examine students' comprehensive analysis ability. According to the discussion and communication of the students, we got a unified first step plan, which was to test the phase of the sample. However, the results of the experiment surprised everyone, because the fresh samples and the samples placed for six months showed no significant changes. According to our theoretical analysis, the oxidation process occurred from the surface at the nanometer scale, so very limited information was detected by XRD tests. At this time, the students were also confused about the next analysis method. The teacher suggested that the students could consider the change of microstructures and surfaces, and many samples were changed on the surfaces, which affected the properties of the whole sample. Then, the students began to use surface analysis methods such as IR, Raman, TEM, XPS, etc. Meanwhile, unexpected oxidation degree of the surfaces was observed for fresh MC samples that were exposed to the air for different time (fresh, one day, one week and one quarter). The relevant test graph is shown in Figure 1b. Comprehensive information for the MOx (HfO_2, ZrO_2, Nb_2O_5, Ta_2O_5 and TiO_2) film and MC (HfC, NbC, ZrC, TaC and TiC) interlayer could be detected by the XPS tests. Meanwhile, the HRTEM and Raman tests could give some additional or supporting information for XPS tests, when the samples were exposed to the air for longer time (one week or one quarter). The oxidation progress of the surfaces of HfC, NbC, ZrC, TaC and TiC took place in air under the ambient temperature condition and the oxidation film was about 2-5 nm [5].

Through a series of discussion activities and test analysis, the enterprise production case was finally formed, and the students also broadened their horizons. The production cases of enterprises are relatively complicated, and students cannot find similar answers from books, thus improving students' understanding and application of basic knowledge. The challenging and unknown enterprise cases greatly enrich the teaching resources. Moreover, the implementation of production and teaching cases in enterprises can enhance students' understanding and application of theoretical knowledge, guiding students to change their emphasis from knowledge to ability.

3.3 Laboratory Ability Verification Project Cases

Laboratory competence verification is an activity to determine laboratory competence through the comparison of test results between laboratories. It is an important means to evaluate laboratory technical competence internationally and can provide effective external quality assurance for laboratories. Ability verification is the necessary means and important basis to prove the technical ability of laboratory. University laboratory comparison activities by the center for scientific research and development in higher education institutes are very authoritative, and they can be called the national test center technical ability of the big competition. The national metrology certification college review group organized the national university inspection and testing institutions to implement during the university laboratory comparison activities. At present, inter-laboratory comparison has become an important means for universities to improve the level of laboratory management and technology. The Center for Materials Research and Analysis is a member of the China Metrology Certification (CMA). In every year, it must participate in numerous university review groups or the national certification and accreditation supervision and administration of the laboratory comparison plan. Therefore, numerous highly difficult cases from university laboratory comparison activities are collected.

In a laboratory capability verification project, the unit participated in the phase and morphology assessment of an unknown sample. The conclusion of this assessment was satisfactory, which indicated that the test results were accurate and reliable. The relevant test results are shown in Figure 2a. Three test methods (XRD, EDS and SEM) were used in this examination. Through XRD analysis, the main phases in the sample were yttrium oxide with cube core structure (Y_2O_3, S.G. 206), alumina with tripartite R-core structure (Al_2O_3, S.G. 167) and metal nickel with cubic core structure (Ni, S.G. 225). The microscopic analysis of unknown powder by SEM showed that there were three microscopic morphologies (angular particles, dendritic particles and fine agglomerated particles) of the powder particles at low magnification. After further magnification, the surface of the multi-angular particles showed porous sponge-like, worm-like or island-like structures. The dendritic particles had branched, chrysanthemum-like or coral-like structures. The fine agglomerated particles showed a rectangular crystal with complete crystal, and the crystal size was relatively uniform. In order to identify the phases in the unknown powder, EDS analysis was carried out on three kinds of microscopic particles. The angular particles were composed of aluminum (Al) and oxygen (O) elements. Dendritic particles were composed of nickel (Ni) elements. The fine agglomerated particles consisted of yttrium (Y) and oxygen (O) elements.

(a) (b)

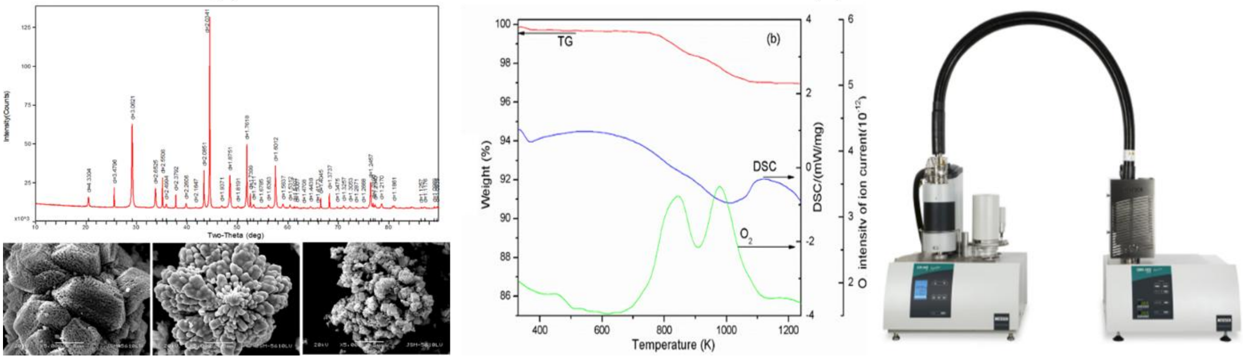


Fig. 2 (a) Phase and morphology analysis and (b) TG-DSC-MS analysis results.

The difficulty of this examination was that the diffraction peaks of phases were very close, so it was difficult to distinguish. A single XRD test method was difficult to get accurate results. With the help of EDS and SEM, the teacher got accurate answers. This kind of laboratory ability verification project must have a difficult point (test point), requiring excellent testing technology accumulation in order to deal with this difficult problem. Therefore, the test analysis ability of students could be significantly improved through this kind of laboratory ability verification project cases.

3.4 Cutting-edge Cases of Hyphenated Technology

Basic theoretical case is designed with the mixture of industrial raw materials. This case is mainly concerned with component analysis, and according to the various testing techniques covered in this course, EDS (Energy Dispersive X-Ray Spectrometer), XRF and XPS can quickly detect compositions of samples. As the level of research continues to improve and research objects become more and more complex, it is difficult for a single testing method to characterize sample information in depth, and abundant hyphenated technologies have gradually emerged and been widely used. Aiming at the testing technology cases of hyphenated technology, this course mainly introduces the hyphenated technology of IR, Raman, TA, DTA, DSC, mass spectrometer (MS) and other equipment. Thermogravimetric-infrared technology (TGA-FTIR) uses purge gas (usually nitrogen or air) to remove the volatile components or decomposition products produced during the thermal weight loss process at a constant high temperature. TGA-FTIR can determine the component structure of the escaped gas through infrared detection and analysis. By using thermal analysis and infrared spectroscopy technology to detect and analyze the gases released during the thermal decomposition of RDF, we could understand the release of gases during the thermal decomposition of RDF and speculate on the possible reaction mechanism of the substance. Pyrolysis gas chromatography-mass spectrometry (Py-GC-MS) technology combines thermal pyrolysis technology with gas chromatography-mass spectrometry technology. Py-GC-MS is not only suitable for the qualitative detection of conventional polymers, but also for the analysis of insoluble and complex organic substances, especially for the analysis of complex polymer substances. The working principle of the TG-DSC/MS is as follows. The sample in the TA instrument is heated to cause decomposition, and the generated gas products are blown into the mass spectrometer through the connector using purge gas. It is possible to directly analyze the molecular weight of the gaseous substances produced. Thermogravimetric and mass spectrometric systems use thermogravimetric analyzers and mass spectrometers respectively. Since there were amorphous manganese oxides and a small amount of other crystalline manganese oxides in the sample, XRD was difficult to detect, and other types of tests were also difficult to accurately obtain the specific types of manganese oxides in the sample [6]. This teaching case uses TG-DSC-MS equipment to solve the problem of analyzing species in mixed manganese oxides.

The TG-DSC-MS was conducted in nitrogen to observe the change in the weight of catalysts, since detecting oxygen coming from manganese oxides due to its decomposition could identify the species of manganese oxides as shown in Figure 2b. Two reactions (Mn_3O_4/Mn_2O_3 and Mn_2O_3/MnO_2) took place, causing that the O_2 was detected at about 800 K and 1050 K. Based on the O2 bimodal distribution, the species and amount of manganese oxides could be obtained. Compared with XRD studies, Mn_2O_3 and MnO_2 had good distribution and formation of particles with very small sizes, existing with amorphous states. Therefore, different MnO_x were identified by thermodynamic parameters and TG-DSC-MS studies.

In addition, there are other related studies that have used joint use to solve some problems and made some innovative discoveries. Hazekamp used a Raman-SEM hybrid system to determine the specific location of polyethylene balls, and then used Raman to obtain chemical and molecular information about the balls [7]. FTIR-Raman coupling technology was used to study the mechanism of the indium oxide ethanol gas sensor by Sanze, and the effects of gas environment, temperature, and different adsorbates on the sensor surface were clearly examined [8]. Therefore, the joint use can obtain more information to provide some new insights. According to the cutting-edge cases of hyphenated technology, students can obtain the in-depth understanding of material testing techniques to solve technical problem from the enterprise production field and scientific research field.

Based on the designing scheme of the basic theoretical cases, enterprise production cases, laboratory ability verification project cases, and cutting-edge cases of combined technology, lots of practical and effective cases are collected [9]. In order to build the Information-based educational resources, the digital case database are built to broaden the channels and ways for students to study the course and help students realize the new learning mode of learning at any time, flexible learning and long-term learning. Students can discuss the related cases online, so they can study curriculum theory in depth. Furthermore, the digital case database are opened for the students who select the course, and the case teaching is carried out during the classroom teaching.

Table 1. Contrastive analysis before and after case teaching

Year	2021	2022	
Full score of the test	100	100	
Mean score	76	80	
Percentage of students achieving 70 or more	80.0%	93.0%	

As shown in Table 1, prior to the case teaching session, the mean score was 76 when the full score of the test was 100. The percentage of students achieving 70 or more was 80%. Meanwhile, the mean score was 80 when the full score of the test was 100 after the case teaching program was carried out during the class. The percentage of students achieving 70 or more was 93%. According to the above results, it can be concluded that the test scores have been significantly improved after the implementing of the case teaching.

Table 2. Satisfaction survey before and after case teaching

Year	2021	2022	
Course satisfaction	97.4%	97.6%	
Percentage of survey engagement	100%	100%	
Percentage of students giving 90 or more	88.6%	94.1%	

Moreover, as shown in Table 2, after completing case teaching session, the course satisfaction survey was conducted to evaluate the student satisfaction. The course satisfaction raised from 97.4% to 97.6%, although this course was always very attractive for the students. The percentage of survey engagement was 100% in both years, illustrating that students were willing to participate in the survey to promote curriculum construction. Meanwhile, the percentage of students giving 90 or more raised from 88.6% to 94.1%, suggesting that more students were satisfied with case teaching for the course of modern testing technology of materials.

4. Summary

This study has independently designed basic theoretical cases, collect enterprise production cases, refine laboratory proficiency verification project cases, and organize cases of the latest hyphenated technology to build the digital case database, achieving in-depth study and application of the basic principles, comprehensive analysis, and innovative development of testing technology. The teachers participating in this project have rich experience in instrument operation and data analysis, and can design original teaching cases with strong professionalism, wide application range, and strong innovation to promote students' in-depth understanding of testing principles. The testing center has well-designed university comparison project resources with clear analysis methods, rigorous processes and accurate conclusions. These cases put forward higher requirements and challenges for students' comprehensive analysis ability, and can improve students' ability to use knowledge to solve complex engineering problems. Analyzing practical problems in production practice and exploring the latest testing technology can stimulate students' interest and creativity, prompt students to provide solutions for developing or improving testing methods, thereby improving students' comprehensive innovation capabilities and job matching.

The biggest feature of case teaching method lies in its authenticity. Because teaching cases are typical events that happen in real situations, they are practical and persuasive, more specific, visual and easy to learn. During the case teaching processing, teachers play the role of guides and designers to guide students to think, discuss and summarize, and ensure the progress of classroom teaching as planned, which highlights the principal position of students in teaching and fully mobilizes their learning initiative. Meanwhile, the digital case database can help the students achieve the In-depth study and discussion online. Furthermore, according to the survey, more students were satisfied with case teaching for the course of modern testing technology of materials, improving their test scores.

Acknowledgements

This work was financially supported by the Cooperative Education Program of Ministry of Education (230804382134739) and the 2023 program of graduate education and teaching reform at Wuhan University of Technology (construction and research of the teaching case base for the course named contemporary analytical techniques for material characterization).

References

[1] Liping, Xiang Hongliang, Liu Dong, He Fushan. The application of case teaching method in material analysis test method. Educational Informatization Form, 2019, (12): 7-8.

[2] Kevin M Bonney. Case study teaching method improves student performance and perceptions of learning gains. Journal of Microbiology and Biology Education, 2015, 16: 21-28.

[3] Susan F McLean. Case-based learning and its application in medical and health-care fields: A review of worldwide literature. Journal of Medical Education and Curricular Development, 2016, l 3: 39-49.

[4] Neda M. Bassir Kazeruni, Andre Laboy, Henry Hess. Designing a hybrid engineering course combining case-based and lecture-based teaching. 2018 40th Annual International Conference of the IEEE Engineering in Medicine and Biology Society (EMBC). Honolulu, HI, USA, 2018, 1636-1639.

[5] Fang De, Wang Chuan, Lv Caihong, Lv Yinmei, Huang Guanlin, Yang Jingyi, Pan Shengxing. XPS studies of surface oxidation of metal carbides. Fullerenes Nanotubes and Carbon Nanostructures, 2022, 30: 718-726.

[6] Xie Junlin, Fang De, He Feng, Chen Junfu, Fu Zhengbing, Chen Xiaolin. Performance and mechanism about MnO_x species included in MnO_x/TiO_2 catalysts for SCR at low temperature. Catalysis Communications, 2012, 28: 77-81.

[7] Johan Hazekamp, Maureen G. Vyvyan C. Howard, Aart A. Van Apeldoorn, Cees Otto. The feasibility of cryo In-SEM Raman microspectroscopy. Journal of Microscopy, 2011, 244: 122-128.

[8] Sandra Sanze, Christian Hess. Ethanol gas sensing by indium oxide: An operando spectroscopic Raman-FTIR study. Journal of Physical Chemistry C, 2014, 118: 25603-25613.

[9] Faiez Alani, Rehmat Grewal. Comparison between in-person versus virtual case-based learning for an upper year course in engineering technology education. International Journal of Engineering Education, 2024, 40: 499-510.