Written review by the official reviewer
on the dissertation of Daniel Mekonnen Zellele
entitled "Electrochemical synthesis and functional properties of metal and alloy-based composition coatings"
submitted for the degree of Doctor of Philosophy (PhD)
in the educational program "8D07110 – Materials Science and Technology of New Materials"

No.	Criteria	Compliance with the Criteria (underline one option)	Justification for the official reviewer's position (Comments should be italicized)
1	The topic of the dissertation (as of the date of its approval) corresponds to the priority areas of scientific development and/or state programs.	1.1 Compliance with priority areas of scientific development or state programs: **1) The dissertation was completed within the framework of a project or target program financed from the state budget (indicate the name and number of the project or program)** 2) The dissertation was completed within the framework of another state program (indicate the name of the program) 3) The dissertation corresponds to a priority area of scientific development approved by the Higher Scientific and Technical Commission under the Government of the Republic of Kazakhstan (specify the area)	The research conducted in this dissertation was carried out within a grant-supported project funded by the Ministry of Science and Higher Education of the Republic of Kazakhstan for the period 2024–2026 (IRN No. AP23484310), titled "Composite coatings from binary and ternary alloys and hetero-oxide compounds with tailored functional properties," under the priority direction "Energy, Advanced Materials, and Transport." As part of this project, applied research activities were carried out at Al-Farabi Kazakh National University and Wroclaw University of Science and Technology in Poland. The dissertation corresponds to the priority field of scientific development approved by the Higher Scientific and Technical Commission under the Government of the Republic of Kazakhstan: "Energy, Advanced Materials, and Transport."
2	Significance for science	The work **makes**/does not make a significant contribution to science, and its importance is **well demonstrated**/not demonstrated.	This dissertation represents a substantial contribution to the development of materials, science and technology. The investigations carried out produced new experimental data on how coating structure and properties depend on processing parameters. The identified optimal processing conditions enable the formation of coatings with controlled structure and phase composition, allowing the attainment of the required set of surface performance and functional properties. The findings of this research support progress in the production of protective coatings on various substrates, providing a basis for further studies and broadening the potential for practical application in modern technological fields.

3	Principle of independence	Level of independence: 1) **High** 2) Medium 3) Low 4) No Independence	The dissertation was carried out with a high degree of independence. The doctoral student was actively involved in every stage of the research, including planning and performing experiments, as well as processing, analyzing, and interpreting the obtained results. His scientific publications, in which he is listed as the first author, serve as evidence of the doctoral student's independence.
4	Principle of internal unity	4.1 Justification of the relevance of the dissertation: 1) **Justified** 2) Partially justified 3) Not justified	The fabrication and investigation of protective coatings on steel substrates is of considerable interest due to their broad application in construction and mechanical components. Enhancing performance characteristics such as corrosion resistance and microhardness through electrodeposition can help extend their range of use. The application of promising strategies, including optimization of electrodeposition temperature and current density, enables energy efficiency while ensuring high growth rates and quality of the resulting coatings. The study results confirm the feasibility of employing electrodeposition on steel substrates as an effective alternative to other techniques. The relevance and scientific importance of the research are supported by publications in highly ranked scientific journals.
		4.2 Does the dissertation's content reflect the dissertation's topic: 1) **Reflects** 2) Partially reflects 3) Does not reflect	The dissertation content fully corresponds to the stated topic. It is composed of four sections that consistently address the theoretical background, experimental investigations, and a synthesis of the results. Each section is logically connected to the overall aim and contributes to addressing the scientific research problem. The first section is dedicated to a review of existing literature on electrodeposition and its processes. It also considers the effect of various electrodeposition parameters on the structural features and final properties of coatings. The second section outlines the experimental methodologies and describes the operating principles of the different instruments used to analyze the structural

			and functional properties of the coatings. The third section, consisting of three main parts, presents and provides a detailed analysis of the experimental results, identifies the key relationships governing the structure and properties of the obtained coatings, and discusses the findings in terms of interpretation and comparison with both domestic and international literature.
		4.3 The aim and objectives correspond to the dissertation topic. 1) **Correspond** 2) Partially correspond 3) Do not correspond	The aim and objectives of the study are fully aligned with the dissertation topic. The main research goal has been achieved, and all stated objectives have been successfully fulfilled. These objectives were accomplished through the synthesis of chromium–graphene, nickel–copper, and cobalt–vanadium composite coatings using the electrodeposition method, along with the investigation of their electrochemical and mechanical properties.
		4.4 All sections and provisions of the dissertation are logically interconnected: 1) **completely interconnected** 2) partially interconnected 3) no interconnection	The dissertation has a well-defined internal structure, where all sections and scientific statements are logically interconnected. The work comprises a comprehensive review of literature data, the fabrication of metal- and alloy-based composite coatings, followed by the investigation of their structural characteristics, as well as their electrochemical and mechanical properties, and the formulation of corresponding provisions for defense.
		4.5 The author's proposed new solutions (principles, methods) are substantiated and evaluated in comparison with existing solutions: 1) **A critical analysis is present** 2) The analysis is partial 3) The analysis does not represent the author's own opinions but rather quotes from other authors 4) No analysis	This work provides a critical analysis of the electrodeposition method for synthesizing functional metal- and alloy-based composite coatings. The proposed strategies for optimizing processing parameters to attain high-performance functional characteristics and process efficiency are regarded among the most effective solutions, enabling a substantial reduction in energy consumption while preserving improved electrochemical and mechanical performance. The study thoroughly evaluates the advantages of the applied approaches in comparison with existing methods, as confirmed by the high-performance indicators obtained in this research.

5	Principle of scientific novelty	5.1 Are the scientific results and findings new? 1) **Completely new** 2) Partially new (25-75% new) 3) Not new (less than 25% new)	The scientific results and conclusions presented in this dissertation are novel. The study led to the establishment of optimized processing conditions that ensure specific structural and operational characteristics of substrate surfaces through electrodeposition, a widely used method for producing functional coatings and regarded as superior to many alternative techniques for various scientific, technological, and economic reasons, particularly for functional and protective applications. In addition to improvements in thickness, microstructure, composition, and morphology, enhanced mechanical properties and corrosion resistance of the coatings were demonstrated. Moreover, the reliability of the obtained data is supported by comparisons with literature sources and findings from similar studies, confirming the high scientific and practical significance of this work.
		5.2 Are the dissertation's conclusions new? 1) **Completely new** 2) Partially new (25-75% new) 3) Not new (less than 25% new)	The results of this dissertation are entirely novel and make a significant contribution to the advancement of coating technologies. 1 A method for the electrodeposition of chromium-based composite coatings reinforced with graphene was developed. It was demonstrated that the quantitative content of the dispersed phase in the chromium matrix can be controlled by adjusting the electrochemical synthesis temperature. The crystal size of the synthesized composite electrolytic coatings (CEC) can be intentionally regulated by varying the synthesis parameters, producing a nanocrystalline structure at 288–303 K, a microcrystalline structure at 316–320 K, and a cracked, microporous structure at 301–305 K. 2 The microstructure of CEC based on binary Ni–Cu alloys was analyzed using AFM, SEM, and XRD. Experimental results showed that at a current density of 40 ± 1.5 mA/cm², a nanocrystalline, homogeneous semi-

			spheroidal morphology with minimal porosity and the highest structural integrity among the tested conditions is formed. The Ni–Cu coatings display a strong (111) preferred orientation and no oxide phases, confirming a stable nanocrystalline growth mechanism under the selected deposition parameters. 3 A pulsed electrolysis mechanism enhancing vanadium incorporation into Co–V alloys was established. The study experimentally validated a new Co–V alloy formation mechanism during pulsed electrodeposition, showing that vanadium incorporation is significantly increased during the pause period due to chemical reduction of vanadyl species by adsorbed hydrogen atoms. Systematic variation of current amplitude, pulse/pause duration, and electrolyte composition resulted in a twofold increase in vanadium content and a current efficiency of 80%, significantly exceeding direct-current deposition values. 4 Citrate-based electrolytes and optimized deposition regimes for Co–V coatings with improved corrosion resistance were developed. New citrate electrolyte compositions and electrodeposition modes for binary Co–V coatings were designed and validated, ensuring stable coating formation, better alloy homogeneity, and enhanced corrosion resistance. It was shown that higher vanadium content shifts the corrosion potential and decreases corrosion current density, confirming the protective effect of vanadium-induced oxide–hydroxide layers. These findings expand the technological capabilities for producing durable, chemically resistant cobalt-based alloy coatings.
		5.3 Technical, technological, economic, or management decisions are new and justified: 1) **Completely new**	The technical, technological, and economic solutions presented in this dissertation are entirely novel and well substantiated. Enhanced overall efficiency and productivity in the electrodeposition process are achieved

		2) Partially new (25-75% are new) 3) Not new (less than 25% are new)	by employing optimal electrolyte concentrations and process parameters in both direct-current and pulsed-current modes, representing a highly promising approach for energy conservation in coating technology. The improved mechanical and functional properties of the coatings, obtained through the addition of alloying elements to the electrolyte, contribute to lower production costs and increased technical and economic efficiency. The study results confirm the feasibility of using electrodeposition on various metals and alloys as an effective alternative to conventional methods, broadening its potential for practical application across multiple industries.
6	Validity of the main conclusions	All main conclusions are **based**/not based on scientifically sound evidence or are sufficiently well justified (for qualitative research and fields in the arts and humanities)	The primary conclusions of the dissertation are supported by scientific evidence obtained from experimental studies and are fully consistent with established theoretical and practical principles in materials science.
7	The main provisions submitted for defense	It is necessary to answer the following questions for each statement separately: 7.1 Is the statement proven? 1) **Proven** 2) Mostly Proven 3) Mostly Not Proven 4) Not Proven 5) In its current formulation, it is impossible to verify whether the statement is proven 7.2 Is the statement trivial? 1) Yes 2) **No** 3) In its current formulation, it is impossible to verify the triviality of the statement 7.3 Is the statement novel? 1) **Yes**	Provision 1: Chromium-graphene composition coatings were electrodeposited on low carbon steel at deposition temperatures of 15 °C, 30 °C, 45 °C, and 60 °C (±2 °C at each deposition temperature) to study the temperature-dependent incorporation of graphene into the chromium matrix and its effect on morphology, mechanical, and corrosion properties. It was revealed that coatings obtained at a deposition temperature of 45 ^{0}C (±2 °C) showed the highest carbon content, coarser morphology, refined grain size, superior hardness and corrosion resistance properties, indicating a synergetic barrier and reinforcing effect of graphene within the chromium matrix. In contrast, relatively flat coatings were obtained at 15 and 30 °C (±2 °C), while coating thickness increased and degradation occurred, accompanied by reduced carbon incorporation at 60 °C (±2 °C). 1) proven 2) it is not trivial

		2) No 3) In its current formulation, it is impossible to verify the novelty of the statement 7.4 Level of Applicability: 1) Narrow 2) Medium 3) **Broad** 4) In its current formulation, it is impossible to verify the level of applicability of the statement 7.5 Is it proven in the article? 1) **Yes** 2) No 3) In its current formulation, it is impossible to verify whether the statement is proven in the article	3) it is new 4) its scope of application is broad 5) it is proven in the article Provision 2: The microstructural evolution of Ni-Cu coatings electrodeposited on AISI 304 at high current densities (20-40 mA/cm²) was systematically analyzed, revealing a strong correlation between deposition parameters and the formation of dense, nanocrystalline, low-porosity coatings. An optimal nanocrystalline structure with an average coherent domain size of ~17 nm was achieved at 40 mA/cm², as confirmed by combined SEM, EDS, XRD, and AFM analyses, indicating a favorable balance between nucleation and growth kinetics in a Watts bath. A unified structure-morphology relationship was established, showing that (111)-oriented nanograins assemble into micron-scale semi-spheroid surface features, providing a mechanistic foundation for tailoring the mechanical and corrosion performance of Ni-Cu coated AISI 304. 1) proven 2) it is not trivial 3) it is new 4) its scope of application is broad 5) it is proven in the article Provision 3: A new pulse-electrodeposition strategy for Co-V alloys was developed, demonstrating that controlled pulse parameters (current amplitude, pulse/pause ratio) significantly enhance vanadium incorporation-up to 1.5 wt.%-beyond what is achievable in stationary mode. The mechanism of additional chemical reduction of vanadium during the pause period was experimentally verified, establishing the role of adsorbed hydrogen atoms in increasing vanadium co-deposition and improving current efficiency (up to 80%). A correlation between vanadium content, microstructure evolution, and functional performance was established, showing that higher

			vanadium incorporation leads to formation of globular and mixed-phase microstructures, increased microhardness (by 100-200 kg/mm² vs. pure Co), reduced hydrogen evolution overpotential (approaching Pt level kinetics), and improved corrosion resistance property in alkaline media. 1) proven 2) it is not trivial 3) it is new 4) its scope of application is broad 5) it is proven in the article
8	Principle of reliability: Reliability of sources and information provided	8.1 Is the choice of methodology justified or is the methodology described in sufficient detail? 1) **Yes** 2) No	The research methodology was selected according to the principles of synthesizing metal- and alloy-based composite coatings. Electrodeposition, a highly promising technique, was employed to fabricate the coatings.
		8.2 The results of the dissertation were obtained using modern scientific research methods and data processing and interpretation techniques using computer technology: 1) **Yes** 2) No	The results of this dissertation were achieved using modern equipment and research techniques, with data processing performed using the latest software tools.
		8.3 Theoretical conclusions, models, identified relationships, and patterns have been proven and confirmed by experimental research (for areas of training in pedagogical sciences, the results have been proven on the basis of a pedagogical experiment): 1) **Yes** 2) No	The theoretical conclusions, models, and identified relationships and patterns are validated and supported by experimental studies conducted using a comprehensive set of modern, state-of-the-art methods, equipment, and software.
		8.4 Important claims are **supported**/partially supported/not supported by references to relevant and reliable scientific literature.	The main claims of the dissertation are substantiated with references to relevant and credible scientific literature. The doctoral candidate cites up-to-date research and publications in materials science and surface engineering,

			demonstrating a comprehensive understanding of the scientific context and the current state of the field.
		8.5 The reference sources used are **sufficient**/not sufficient for a literature review.	The dissertation includes 216 references, which is adequate to provide a thorough and comprehensive literature review.
9	Principle of practical value	9.1 Does the dissertation have theoretical significance? 1) **Yes** **2)** No	The dissertation does not have a primary theoretical purpose.
		9.2 The dissertation has practical significance and there is a high probability of practical application of the results obtained: 1) **Yes** 2) No	This dissertation holds considerable practical significance, as the results can be applied to the development of technologies for producing metal- and alloy-based functional coatings. These findings enhance corrosion resistance, improve mechanical strength, and ensure high surface performance. The practical applications of this research extend to mechanical engineering, aviation, medical instrumentation, electronics, and other related industries.
		9.3 Are the practical recommendations new? 1) **Completely new** 2) Partially new (25–75% are new) 3) Not new (less than 25% are new)	The practical recommendations presented in this dissertation are novel and possess considerable practical value. The findings demonstrate that electrodeposited functional coatings can be effectively applied to improve surface, mechanical, and corrosion-resistant properties.
10	Quality of writing and design	Quality of academic writing: 1) **High** 2) Average 3) Below average 4) Low	The academic writing in this dissertation is of high quality. The text is clear, logical, and coherent, making the scientific concepts and conclusions easily understandable. The dissertation adheres to academic standards, which enhances both its scientific value and accessibility to readers. A small number of typographical errors does not diminish the overall quality of the work.
11	Comments on the dissertation	While the dissertation presents a comprehensive experimental investigation of electrochemically deposited Cr–graphene, Ni-Cu, and Co-V coatings with a strong focus on corrosion and mechanical properties, the following remarks are offered for consideration:	

		1. In addition to corrosion and mechanical property evaluations, further tribological investigations would be valuable to better understand the wear behavior of the coatings and to provide a more complete functional characterization of the studied systems. 2. Conducting real-time testing of the coated materials in practical applications, such as structural components or construction elements, would provide deeper insight into their long-term applicability, performance, and reliability under service conditions. 3. The coatings have been produced at a laboratory scale; therefore, scaling up the production process would be essential to enable comprehensive cost analysis and to assess the environmental implications of the proposed electrodeposition technologies more thoroughly. These remarks are intended as constructive recommendations and do not diminish the scientific value of the work presented.
12	The scientific level of the doctoral student's articles on the research topic (in the case of defending a dissertation in the form of a series of articles, official reviewers' comment on the scientific level of each article of the doctoral student on the research topic)	The scientific quality of the doctoral candidate's articles meets the standards required for publications related to the dissertation research. These articles were published in peer-reviewed journals, including those indexed in Scopus, Web of Science, and listed by the Quality Assurance Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan. The publications present the key theoretical principles, methodological approaches, and research findings, demonstrating the candidate's independent scientific approach as well as the relevance and novelty of the results.
13	Decision of the official reviewer (in accordance with paragraph 28 of current Standard Regulations)	The dissertation fully satisfies all the requirements for the Doctor of Philosophy (PhD) degree, and Zellele Daniel Mekonnen is deserving of the PhD degree in the educational program 8D07110 – Materials Science and Technology of New Materials.

Official Reviewer
Candidate of Chemical Sciences (PhD),
Institute of Metallurgy and Ore Beneficiation,
Satbayev University

Dametken Yedilovna Fischer

Құқық және кадрлармен жұмыс Департаменті

Қолы/подпись Фишер Д.Е.
растаймын / заверяю
Ғылыми хатшы / Ученый секретарь
«Металлургия және кен байыту институты» АҚ
«20» 04 2026 ж.