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 presented in this dissertation was carried out within the framework of a grant-funded project of the Ministry of Science and Higher Education of the Republic of Kazakhstan for 2024–2026, IRN No. AP23484310, "Composite coatings from binary and ternary alloys and hetero-oxide compounds with tailored functional properties", under the priority area "Energy, Advanced Materials, and Transport". Within this project, applied research was conducted in Al-Farabi Kazakh National University and Wroclaw University of Science and Technology, Poland. This dissertation is in line with the priority area 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 makes a significant contribution to the advancement of materials science and technology. The studies conducted yielded new experimental data on the relationship between the structure and properties of coatings depending on processing parameters. The developed optimal processing conditions ensure the formation of coatings with a controlled structure and phase composition, enabling the achievement of the desired set of surface performance and functional properties. The results of the research contribute to advances in the field of producing protective coatings on the surface of various substrates providing a foundation for further research and expand the prospects 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 completed with full level of independence. The doctoral student actively participated in all stages of the research, from planning and conducting experiments to processing, analyzing, and summarizing the obtained results. His scientific publications, for which he is the first author, can be considered as an indicator 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 production and study of the properties of protective coatings on steel substrates is of significant interest due to the wide range of its applications in construction and machinery parts. Improving the performance characteristics like corrosion resistance and microhardness of coatings using electrodeposition method can contribute to expanding their application. The use of promising approaches, such as optimizing the electrodeposition temperature and current density allows for energy savings and ensures high growth and quality of the resulting coatings. The results of the study substantiate the feasibility of using electrodeposition on steel substrates as an effective alternative to other methods. The relevance and scientific significance of the conducted research are confirmed by publications presented 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 content of the dissertation is fully consistent with the stated topic. It consists of four sections, sequentially examining the theoretical foundations, experimental studies, and a summary of the results. Each section is logically linked to the overall objective and contributes to the scientific research question. The first section is devoted to a review of available literature on electrodeposition and its processes. The influence of various electrodeposition processing parameters on the structural characteristics and final properties of coatings is also discussed. The second section presents the experimental research methods, describes the operating principle of the various

			tools used for analyzing structural and functional properties of coatings. The third section. Consisting of three main subjects, presents and thoroughly analyzes the experimental results, identifies key patterns determining the structure and properties of the resulting coatings, and discusses the data obtained in the context of their interpretation and comparison with domestic and international available 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 consistent with the topic of the dissertation. The research objective has been achieved, and all objectives have been fully addressed. The objectives were fully addressed by synthesizing chromium-graphene, nickel-copper, and cobalt-vanadium composite coatings using electrodeposition method and studying the electrochemical and mechanical properties of the coatings.
		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 clear internal structure, in which all sections and scientific propositions are logically interconnected. The work includes an extensive analysis of literature data, the production of metal and alloy -based composite coatings, a subsequent study of their structural characteristics, electrochemical and mechanical properties, and the formulation of corresponding propositions 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 presents a critical analysis of the method of electrodeposition to synthesize functional metal and alloy-based composite coatings. The proposed approaches to optimizing processing parameters to achieve high performance functional characteristics and process indicators are considered among the most effective solutions, providing a significant reduction in energy consumption while maintaining enhanced electrochemical and mechanical performance. The study examines in detail the advantages of the applied approaches compared to

			existing methods, which are confirmed by the high-performance indicators achieved in this study.
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 derived from this dissertation are novel. This study resulted in the synthesis of optimized processing conditions that ensure a set of specified structural and operational characteristics of the surface of substrates using electrodeposition, a widely employed method of synthesizing functional coatings and considered superior to many other coating techniques for several scientific, technological, and economic reasons, especially in functional and protective coatings. Besides enhancements in thickness, microstructure, composition and morphology, an improvement in mechanical and corrosion resistance properties of the coatings was demonstrated. Furthermore, the validity of the obtained data is confirmed by comparison with literature sources and the results of similar studies, demonstrating 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 findings of this dissertation are entirely new and represent a significant contribution to the development of coating technologies. 1. A technique for the electrodeposition of chromium-based composite coatings reinforced with graphene has been developed. It has been shown that the quantitative content of the dispersed phase in the chromium matrix can be controlled by varying the temperature of electrochemical synthesis. The crystal size of the synthesized composite electrolytic coatings (CEC) can be purposefully regulated by changing electrochemical synthesis parameters, resulting in the formation of a nanocrystalline structure in the deposition temperature range of 288–303 K, a microcrystalline structure at 316–320 K, and a cracked and microporous structure at 301–305 K. 2. The microstructure of CEC based on binary Ni–Cu alloys was studied using AFM, SEM, and XRD methods.

			It was experimentally demonstrated 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 deposited Ni–Cu coatings exhibit a strong (111) preferred orientation and the absence of oxide phases, confirming a stable nanocrystalline growth mechanism characteristic of the selected deposition parameters. 3. A pulsed electrolysis mechanism providing enhanced incorporation of vanadium into Co–V alloys has been established. A new mechanism of Co–V alloy formation during pulsed electrodeposition has been experimentally substantiated, demonstrating that vanadium incorporation is significantly intensified during the pause period due to the chemical reduction of vanadyl species by adsorbed hydrogen atoms. This mechanism was confirmed through systematic variation of current amplitude, pulse/pause duration, and electrolyte composition, resulting in a twofold increase in vanadium content and a current efficiency reaching 80%, which significantly exceeds the values obtained under direct-current deposition conditions. 4. Citrate-based electrolytes and optimized deposition regimes for Co–V coatings with superior corrosion resistance have been developed. New optimized citrate electrolyte compositions and electrodeposition modes for binary Co–V coatings were designed and validated. The optimized systems ensure stable coating formation, improved alloy homogeneity, and enhanced corrosion resistance. It was shown that increasing the vanadium content shifts the corrosion potential and reduces corrosion current density, confirming the protective effect of vanadium-induced oxide–hydroxide layers. These results expand the technological possibilities for producing durable, chemically resistant cobalt-based alloy coatings.

		5.3 Technical, technological, economic, or management decisions are new and justified: 1) **Completely new** 2) Partially new (25-75% are new) 3) Not new (less than 25% are new)	The technical, technological, and economic solutions proposed in this dissertation are entirely new and well-founded. Increased efficiency and productivity of the electrodeposition process are achieved through the use of optimal concentrations of electrolytic baths, process parameters in both direct current and pulsed current modes, which are considered the most promising approach to energy conservation in electrodeposition coating technology. Improved mechanical and functional properties of coatings, achieved through the introduction of alloying components into the electrolyte contribute to a reduction in production costs and an increase in the technical and economic efficiency of the technology. The results of the study substantiate the feasibility of using electrodeposition method on various metal and alloys as an effective alternative to traditional technologies, expanding the possibilities of its practical application in various 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 main conclusions made in the dissertation are based on scientific evidence obtained during experimental studies and do not contradict theoretical and practical laws 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	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

		3) In its current formulation, it is impossible to verify the triviality of the statement 7.3 Is the statement novel? 1) Yes 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	°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 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 chosen based on the principles of synthesizing metal and alloy-based composite coatings. A promising method, electrodeposition, was used to produce 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 the dissertation work were obtained using modern equipment and research methods, and the processing of the obtained data was carried out using the latest software.
		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	Theoretical conclusions, models, identified relationships and patterns are proven and confirmed by experimental studies obtained through the comprehensive application 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 key claims presented in the dissertation are supported by references to relevant and reliable scientific literature. The doctoral candidate cites current research and publications in the fields of materials science and surface engineering, demonstrating a thorough understanding of the scientific context and the current state of the art.
		8.5 The reference sources used are **sufficient**/not sufficient for a literature review.	The number of references used, 216, is sufficient to form a 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 has significant practical significance, as the results obtained can be used to develop technologies for producing metal and alloy-based functional coatings. These results contribute to increased efficiency in the formation of corrosion, improved mechanical strength, and high surface performance properties. Practical applications of the research findings are possible in mechanical engineering, aviation, medical instrumentation, electronics, and other fields.
		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 proposals presented in this dissertation are new and have significant practical value. The results indicate that electrodeposited functional coatings can be recommended for use as enhancing surface, mechanical and corrosion properties.
10	Quality of writing and design	Quality of academic writing: 1) **High** 2) Average 3) Below average 4) Low	The quality of the academic writing in the dissertation is high. The text is written clearly, logically, and coherently, making the presented scientific ideas and conclusions easy to understand. The dissertation meets academic standards, enhancing its scientific value and accessibility for readers. *A minor number of typos does not detract from the quality of the dissertation.*
11	Comments on the dissertation	1) In Cr-graphene composite coatings, what properties of graphene make it a good candidate for coating on metal substrates?	

		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. 3) The composite coatings were produced at a laboratory scale; therefore, scaling up the production process would be essential to enable comprehensive cost analysis and to assess environmental implications more thoroughly. 4) In addition to corrosion and mechanical property evaluations, further tribological investigations would be valuable to better understand the wear behavior of the coatings.
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 level of the doctoral candidate's articles meets the requirements for publications on the topic of dissertation research. The articles were published in peer-reviewed journals, including journals listed in Scopus, Web of Science, and the list of the Quality Assurance Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan. The publications reflect the main theoretical principles, methodological approaches, and research results, demonstrating the independence of the scientific approach and the relevance and novelty of the findings.
13	Decision of the official reviewer (in accordance with paragraph 28 of current Standard Regulations)	The dissertation work meets all the requirements for dissertations for the degree of Doctor of Philosophy (PhD), and Zellele Daniel Mekonnen deserves to be awarded the PhD degree in the educational program 8D07110 - Materials Science and Technology of New Materials.

Official Reviewer
Candidate of Physical and Mathematical Sciences,
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