

REVIEW

of dissertation for the acquisition of:

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|---|---------------------------------------|
| educational and scientific degree " doctor " | X |
| scientific degree " Doctor of Science " | |
| | the true is indicated by the sign "X" |

Author of the dissertation:

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|-------------------|-------------------|--------|---------------|-----------|----------------------|
| Res. Assist. | | Nikita | Alexandrovich | Lutchenko | Nazarbaev University |
| academic position | scientific degree | name | middle name | last name | workplace |

Topic of the dissertation:

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| Investigation of the possibility of obtaining an ultrafine-grained structure of zirconium alloys by methods of intensive plastic deformation |
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Scientific area:

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|------|---|
| 5 | Technical sciences |
| code | Plastic deformation processing technologies, machines and systems |

Professional area:

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|------|---------------------------------|
| 5.6 | Materials and materials science |
| code | name |

Scientific specialty:

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| Materials science and technology of engineering materials |
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The review was written by:

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|-------------------|-------------------|------|-------------|-----------|-------------|
| Professor | Doctor | Emil | Georgiev | Mihailov | UCTM, Sofia |
| academic position | scientific degree | name | middle name | last name | workplace |

1. Completion of the provided documents:

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|---|----------|---|
| A) The dissertation and the competition documents are in full compliance with the Regulations. | 4 points | X |
| B) The documents are complete but do not fully comply with the requirements of the Regulations. | 2 points | |

| | | |
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| C) The documents are not completed in accordance with the requirements of the Regulations. | 0 points | |
| | | one of the answers given is marked with the sign "X" |

Missing documents and violated standards must be described if response C is marked.

2. Meeting the minimum requirements under the Regulations:

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|--|-----------|--|
| A) The candidate meets the minimum requirements | 20 points | X |
| B) The candidate doesn't meet the minimum requirements | 0 points | |
| | | one of the answers given is marked with the sign "X" |

It must be filled in if answer B is marked. The publication activity of the candidate is analyzed. The response of the results achieved (quoted) is analyzed.

3. The relevance of the topic of the dissertation:

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|---|----------|--|
| A) The topic is relevant and new (there are no known results on the topic by other authors) | 8 points | |
| B) The topic is relevant and results from other authors are known | 6 points | X |
| C) The topic is not relevant, but results from other authors are known | 2 points | |
| D) The topic is not relevant and no results from other authors are known | 1 point | |
| E) The topic does not correspond to the level of dissertation | 0 points | |
| | | one of the answers given is marked with the sign "X" |

The evaluation of the relevance of the dissertation must be substantiated

One of the main applications of zirconium alloys is in nuclear reactors, where fuel rods are clad with a zirconium alloy to prevent the release of radioactive materials into the coolant. Radial shear rolling combines intensive grain crushing with the ability to process long blanks. This makes the method promising for producing cladding for heat-releasing elements in nuclear reactors. In this method of intense plastic deformation, a complex stress-strain state is formed in the deformation zone of the metal, which helps to create a gradient ultrafine-grained structure and a favourable texture. Therefore, the study of the application of radial shear rolling to zirconium alloys to form an ultrafine-grained structure and improve mechanical properties, texture and radiation resistance is a current scientific and applied task that meets the requirements of modern nuclear energy.

4. Knowledge of the problems, subject of research in the dissertation:

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| A) The doctoral student knows in detail the achievements of other authors on the topic of the dissertation | 8 points | X |
| B) The doctoral student is partially familiar with the achieved results on the topic of the dissertation | 4 points | |
| C) The doctoral student has no prior knowledge of the status of the problems in the dissertation | 0 points | |
| | | one of the answers given is marked with the sign "X" |

The evaluation must be substantiated if answer C is marked.

5. Type of research:

| | | |
|--|----------|--|
| A) Theoretical | 4 points | |
| B) Applied | 4 points | |
| C) Theoretical with application elements | 4 points | X |
| D) It does not correspond to the level of dissertation | 0 points | |
| | | one of the answers given is marked with the sign "X" |

The level of research must be substantiated if answer D is marked.

6. Objectives of the research:

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|---|----------|--|
| A) Realistic and of scientific and / or applied interest | 8 points | X |
| B) Realistic, but not of scientific and / or applied interest | 3 points | |
| C) Unattainable (unrealistic) | 0 points | |
| | | one of the answers given is marked with the sign "X" |

Objectives must be specified. The type of the set objectives must be justified.

The goals set in the dissertation work can be summarized as follows:

- To analyze the modern requirements for structural materials used in the reactor core and to identify the key mechanisms of their degradation under irradiation, as well as to assess the feasibility of applying ultrafine-grained (UFG) structures based on zirconium alloys;
- To summarize and classify the existing methods for obtaining UFG structures and evaluate their applicability to zirconium alloys, taking into account scalability, product geometry, and operating conditions in nuclear reactors;

- To develop a technological scheme of radial-shear rolling (RSR) using finite element modelling (FEM) of the process, to investigate the features and regularities of the stress–strain state of zirconium, and to optimize the geometric and technological parameters of rolling;
- To carry out a series of experimental RSR trials and obtain billets of various diameters for analyzing the evolution of the resulting structure;
- To investigate the microstructural evolution across the cross-section of the billets obtained by RSR using transmission and scanning electron microscopy, including EBSD mapping, and to perform an analysis of texture characteristics (including the calculation of Kearns parameters);
- To study the fine structure of samples produced by radial-shear rolling using high-resolution electron microscopy;
- To obtain data on their mechanical properties;
- To conduct heavy-ion irradiation of the samples in order to simulate damage conditions caused by fission fragments in the reactor core.

The goals and methods are grounded in experimental and theoretical research, aimed at a fundamental understanding of the mastery and application of zirconium alloys in nuclear energy.

7. Methods of research:

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| A) Adequate to research and set objectives | 8 points | X |
| B) Partially appropriate, enabling part of the scientific objectives and / or applications to be achieved | 4 points | |
| C) Inappropriate methods | 0 points | |
| | | one of the answers given is marked with the sign "X" |

Methods must be specified. The type of methods used is justified.

Various methods are used in the development of the research work, including experimental studies, mathematical modeling and data processing.

The goals set in the research are realistic and have been achieved with the complex and adequate application of modern research methods.

The candidate's research has a scientific and scientifically applied nature and is aimed at solving theoretical and real technological problems.

8. Contributions of the dissertation:

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| A) With lasting scientific and / or applied response, they form the basis for new research and applications | 20 points | X |
| B) They are of significant scientific and / or applied interest, complete and / or summarize previous research | 16 points | |
| C) They are of scientific and / or applied interest | 12 points | |
| D) Lack of significant contributions | 8 points | |
| E) Lack of contributions | 0 points | |
| | | one of the answers given is marked with the sign "X" |

Contributions must be specified. The type of results achieved must be justified.

Based on the results of the conducted research, the following contributions can be identified.

Scientific contributions:

1. An original systematic scientific approach to studying the technology of radial-shear rolling as a method of severe plastic deformation of zirconium alloys has been developed and implemented, encompassing numerical modelling, experimental rolling, microstructural analysis, mechanical property evaluation, and radiation-resistance testing. The obtained results are original and have no analogues in the existing scientific literature.
2. The feasibility of successfully applying the Radial-Shear Rolling (RSR) method to the Zr-1%Nb (E110) alloy has been demonstrated. The regularities of ultrafine-grained (UFG) structure formation and evolution, as well as their influence on the mechanical properties of the processed samples, have been established. The obtained data expand the fundamental understanding of the mechanisms of grain-structure formation and texture development in zirconium alloys under RSR.
3. The physical mechanisms responsible for the enhancement of strength and radiation resistance upon reducing the grain size to the ultrafine-grained level have been substantiated. It has been shown that the high density of grain boundaries promotes the formation of a stable structure and improves the overall set of material properties.

Applied Scientific Contributions

A technology for forming an ultrafine-grained structure in zirconium alloys using severe plastic deformation methods—specifically, radial-shear rolling (RSR)—has been developed and experimentally validated, followed by a comprehensive analysis of the structural and performance characteristics of the processed material:

1. A finite-element model of the radial-shear rolling (RSR) process was developed in the DEFORM-3D software. The stress–strain state of the Zr-1%Nb (E110) zirconium alloy billet under conditions of high deformation intensity was analyzed. Optimal rolling parameters—including billet temperature, roll rotation speed, single-pass reduction, and friction coefficient—were determined. The simulation results formed the basis for designing experimental regimes and for model verification.
2. It was experimentally demonstrated that radial-shear rolling, which is characterized by its ability to introduce large plastic strains with a pronounced shear component and vortex-like metal flow within the billet, can be effectively used to obtain an ultrafine-grained structure in the investigated zirconium alloy. This approach enabled an increase in strength characteristics while maintaining an acceptable level of ductility.
3. The evolution of the microstructure across the entire transverse cross-section of the bars was investigated with high spatial detail at all stages of radial-shear rolling, which involves substantial shear deformation and vortex metal flow. Microstructural analysis was performed using scanning electron microscopy (SEM/EBSD) and transmission electron microscopy (TEM):
 - a. The evolution of the microstructure was established, including the distribution of grain sizes, the types of grain boundaries, the presence of substructural elements, and the characteristic features of the resulting texture.
 - b. It was determined that the fraction of the ultrafine-grained (UFG) structure in the transverse cross-section of the billet can reach 35–40% of the outer layer, forming a distinct gradient morphology that combines a UFG state in the peripheral region with a submicrocrystalline or recrystallized structure in the central part.
 - c. Using EBSD analysis, highly detailed microstructure and texture maps were obtained, grain orientations were determined, and Kearns parameters reflecting the degree of material anisotropy were calculated. It was established that RSR processing produces predominantly a radial–basal texture in the core of the bar and an atypical rolling texture characterized by a dominant axial basal component in the surface layers.
4. The mechanical properties of the RSR-processed bars were determined and evaluated through uniaxial tensile testing and microhardness measurements across the transverse cross-section.
 - a. An increase in strength compared to the initial material was established while maintaining sufficient ductility.
 - b. A clear correlation between the local microstructure and microhardness was identified, with regions containing a UFG structure exhibiting the highest values.
5. By comparing the results of equal-channel angular pressing (ECAP) — used as a reference method for producing an ultrafine-grained (UFG) structure — with those obtained via radial-shear rolling (RSR), it was established that RSR enables the formation of a metal structure with a comparable degree of grain refinement while requiring fewer processing passes. At the same time, the resulting structure exhibits a gradient character and a specific, potentially advantageous texture. Moreover, RSR demonstrates superior technological scalability.

6. The feasibility of using RSR for processing zirconium ingots with an as-cast structure was investigated. It was established that the RSR method is capable of effectively closing shrinkage and macro-porosity defects due to the intensified plastic flow within the billet volume. The application of ECAP to the cast structure also demonstrated partial defect closure; however, it requires additional preparatory steps.
7. Radiation damage of the alloy was simulated through heavy-ion irradiation using the DC-60 accelerator. This methodology makes it possible to reproduce effects analogous to those caused by fission fragments in nuclear fuel. It was established that the zirconium alloy with an ultrafine-grained (UFG) structure maintains stable morphology, exhibits moderate radiation-induced hardening without accompanying embrittlement, and preserves a stable Young's modulus, indicating the promising potential of this material for long-term operation in nuclear reactors.
8. The effect of temperature on the stability of the obtained ultrafine-grained (UFG) structure and the mechanical properties was established. It was found that at temperatures up to 400 °C, the structure remains stable and the mechanical properties are retained at a high level. At higher temperatures, signs of recrystallization and grain growth appear, accompanied by a decrease in hardness and strength.

Applied (Practical) Contributions:

1. The feasibility of producing long rods of various diameters from the zirconium alloy using the developed RSR technology has been experimentally confirmed, enabling the fabrication of bars with a gradient structure and a high degree of grain refinement.
2. The proposed technology is applicable to the industrial production of structural materials with improved performance characteristics. The ultrafine-grained structure formed in the surface layers of the billets, combined with the atypical crystallographic texture resulting from RSR, creates new opportunities to develop high-efficiency materials for nuclear energy applications, characterized by enhanced radiation resistance and superior mechanical properties during long-term operation. The data obtained can be used to develop new manufacturing technologies for fuel cladding tubes with extended service life and improved safety.

9. Evaluation of the compliance of the dissertation summary with the dissertation:

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| A) Full compliance | 4 points | X |
| B) Compliance of the main parts | 2 points | |
| C) Lack of compliance of the main parts | 0 points | |
| | | one of the answers given is marked with the sign "X" |

The evaluation must be substantiated if answer C is marked.

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10. Participation of the doctoral student in the achievement of the results of the dissertation:

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| A) The doctoral student has at least an equal participation | 8 points | X |
| B) The doctoral student has secondary participation | 5 points | |
| C) The participation of the doctoral student is unnoticeable | 0 points | |
| | | one of the answers given is marked with the sign "X" |

Critical notes must be provided if one of the items B or C is marked.

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11. Critical notes:

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| A) Lack of critical notes | 8 points | X |
| B) Critical notes of a technical nature | 7 points | |
| C) Critical notes that would partially improve the results achieved | 4 points | |
| D) Significant critical notes | 0 points | |
| | | one of the answers given is marked with the sign "X" |

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| Critical notes must be provided if one of the answers C or D is marked. |
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12. Conclusion

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| A) The evaluation of the dissertation is POSITIVE | This evaluation is assigned to a total number of at least 65 points | X |
| B) The evaluation of the dissertation is NEGATIVE | This evaluation is assigned to a total number below 65 points | |
| | | one of the answers given is marked with the sign "X" |

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| To be filled in at the request of the reviewer |
| Based on the analysis of the dissertation work, the research carried out, the publications and the achieved results and in connection with the fact that the candidate has fulfilled the minimum requirements of the Law on DASRB, PNSZAD and PPNSZAD of UCTM for acquiring the educational and scientific degree "Doctor", I give a positive assessment of his activities and allow myself to propose to the esteemed scientific jury to accept and evaluate positively the dissertation work, the publications and the obtained results and to award Eng. Nikita Alexandrovich Lutchenko the scientific degree "Doctor", in the scientific specialty "Materials Science and Technology of Mechanical Engineering Materials", professional direction 5.6. Materials and Materials Science, field of higher education 5. Technical Sciences. |

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| 12.02.2026 | The review was written by: | |
| date | Emil Georgiev Mihailov | signature |