REVIEW

of a dissertation for the acquisition of an educational and scientific degree "Doctor"

Author of the dissertation: **Mag. Eng. Plamen Ivanov Stoyanov**

Title of the dissertation: "**Influence of deep cryogenic treatment on the structure and properties of thermally hardened and thermally non-hardenable aluminum alloys**"

in the professional field 5.1 "Mechanical Engineering" and the doctoral program "Technology of machine-building materials"

Prepared by: **Prof. Dr. Eng. Stoyan Dimitrov Slavov**, Technical University of Varna

(Appointed as a reviewer, according to Order No 774/20.11.2025 of the Rector of the Technical University of Varna)

1. General characteristics of the dissertation

The dissertation is formed into an introduction, four chapters, an assessment of the possibilities for industrial application of deep cryogenic processing, a list of contributions, a list of publications of the doctoral student on the dissertation, a list of participation in research projects and a bibliography. Separately, a declaration of originality of the submitted work was provided and signed by the doctoral student. The total volume of the dissertation is 186 pages, including 119 figures and 52 tables. The list of references contains 193 literary sources, the majority of which are from the last 10 years and in English.

The dissertation is focused on the study of the impact of deep cryogenic treatment (DCT) on the microstructure, mechanical and operational properties of both thermally hardened and non-thermally hardened aluminum alloys. The study aims to identify optimal conditions for DCTs and to clarify the mechanisms behind the observed changes in the characteristics of some aluminum alloys.

Chapter One discusses the structural characteristics and composition of various aluminum alloys, including approaches to their processing and their properties. The focus is on aluminum-magnesium, aluminum-copper, aluminum-silicon alloys, etc., as well as their application in various industrial fields. Their alloying ingredients, their heat treatment conditions and performance characteristics such as mechanical strength, fatigue strength and corrosion resistance are discussed. The temperature ranges for different heat treatments and the conditions for their application are included. The phase states and crystallization processes and the crystallographic structure of alloys are also analyzed. Several finishing technological treatments based on methods of surface plastic deformation and diamond burnishing are also described, as well as the positive effects of their application. The chapter ends with eleven conclusions based on which the purpose and objectives of the current research work are set.

The second chapter is devoted to the various standardized methods (such as EN ISO 6507-1:2006, EN ISO 6892-1:2020, EN ISO 4287, etc.), processing machines and measuring instruments used during research to determine and measure the characteristics of thermally hardened and non-hardening aluminum alloys – the subject of this work. The technological processes for processing with a polishing machine MoPao 260E and the use of reagents such as NaOH+NaF for the preparation of microslices are described.

Chapter Three presents the results obtained after applying the methodologies from Chapter Two. They show that deep cryogenic treatment has a positive effect on the mechanical properties of the studied aluminum alloys AlSi7Mg, AlSiMgMn and AlMg6, especially the microhardness, mechanical strength and improves the corrosion resistance of the studied aluminum alloys, especially when applied after conventional heat treatment. Increased density of dislocations is the most probably reason for these improvements. The X-ray diffraction analysis did not show any new phase formations after DCT in any of the studied alloys. It has been

found that the DCT does not significantly alter the surface' roughness of the samples processed by diamond burnishing, which suggests that it can be used as a final finishing of parts made of the studied aluminum alloys. Regarding the duration of treatment with the DCT, it is clear that the 24-hour residence of the samples leads to better strength properties, and at 48 hours, an improvement in the plasticity of alloys is observed.

Chapter 4 is devoted to research on the mechanical properties (tensile strength Rm, hardness HV1 and relative elongation A) of aluminum alloys of the type AlSi7Mg, AlSiMgMn, and Al-Mg with 4.5% and 6% Mg) under various thermal conditions and the application of deep cryogenic treatment (DCR with a duration of 0, 24, and 48 hours). For each alloy, experimental data were collected and polynomial regression models (including quadratic and with interactions between predictors) were derived for the dependencies Rm = f(HV1) and A = f(HV1, Rm). The models show a relatively high descriptive ability (mean coefficient of determination R^2 about 0.92–0.96 for individual cases), with the coefficients and their confidence intervals accounted for and interpreted statistically. ANOVA analyses and additional diagnostic tests for normality and homoskedasticity of residuals (according to the methods of Shapiro–Wilk, Kolmogorov–Smirnov, Jarque–Bera, etc.) were also carried out to validate the obtained stochastic models, and most of the results obtained support the statistical significance of the specific predictors of the models (mostly HV1 and Rm). The results are visualized by a 3D response surface for the functional dependency A = f(HV1, Rm), which clarifies the nature of their relationship.

As a result, it was found that for the alloy AlSiMgMn (6082), the duration of 48 hours of CTR has an optimal effect of increasing ductility without significantly reducing strength. For AlMg6 and AlMg4.5 alloys, the optimum duration of the DCP is 24 hours at a temperature of -196° C, especially after applying diamond pre-burnishing. DCT is proposed as a final treatment to improve corrosion resistance without affecting the surface's roughness of parts made of these alloys. The chapter concludes with an assessment of the possibilities and recommendations for industrial application of the developed methodology for DCT, combined with a finishing process for mechanical processing - diamond burnishing.

2. Actuality of the problem

Although the dissertation is focused on a field that is not fundamentally new, it remains relevant, as aluminum alloys are widely used in automotive, aviation, construction, maritime and several other industries due to the combination of low weight and good technical performance.

The topic of structural characteristics, alloying, heat treatment and the application of finishing processes based on plastic deformation of the surface layer of aluminum alloys is still very relevant. Especially since in recent years there has been considerable scientific interest in the use of cryogenic treatment of materials. It is applied to increase the strength and ductility of aluminum alloys, improve their resistance to fatigue and corrosion, as well as to obtain a more uniform microstructure for higher mechanical strength. This research area is relevant because it combines traditional processing methods such as surface plastic deformation with a relatively new approach, such as the ACA for increasing the performance characteristics of the aluminum alloys involved in the work, for which there is still no data from similar studies conducted in the literature. In addition, in the available sources regarding cryogenic treatments, there is no data on research carried out with the specific brands of aluminum alloys on which the PhD student focused.

3. Degree of knowledge of the state of the problem and creative interpretation of the literary material.

In this dissertation, a detailed literature review of 63 pages is made based on a large set of literature sources (193 pcs.). The PhD student demonstrates a very good degree of theoretical

and applied knowledge of heat treatment of aluminum alloys. The information is up-to-date, scientifically sound and supplemented with examples and reference data. which controls the formation of reinforcing phases (e.g. $CuAl_2$, Mg_2Si), the redistribution of impurities and secondary phases and the change in the grain structure of aluminum alloys. Particular attention is paid to artificial and natural aging (T6 and T5 states), temperature regimes – in some alloys values around 480–500°C are mentioned, as well as phase transitions during heating and cryogenization. The PhD student shows a high level of understanding of the topic of the dissertation through the inclusion of specific phase diagrams and phase compositions for the aluminum alloys he researches, a description of the effects of heat treatment on their mechanical properties – strength, hardness, plasticity. Numerous post-heat treatment parameter tables are attached for various ENAW-type alloys such as 6082, 7075, etc. At the same time, other technological processes, such as diamond burnishing, are also considered as a different way to further improve the characteristics in the surface layer of the parts. As a result, the purpose of the dissertation and the specific research tasks are defined. This gives me reason to conclude that the degree of knowledge of the PhD student's knowledge of the problems related to the methods for improving the performance characteristics of aluminum alloys is in-depth and at a very good level.

4. Correspondence of the chosen research methodology and the set goal and objectives of the dissertation

The results presented in the dissertation are obtained based on an in-depth analysis of the literature sources. Based on them, a detailed flowchart (see Fig. 2.2) of the materials used, and test methods are presented at the beginning of Chapter 2, including methodologies for measuring hardness, relative elongation, identification of alloy structure, measurement of roughness and corrosion resistance, which are discussed in detail in the following chapters. The methods used are relevant and help to solve the goals and tasks set in the dissertation.

5. Contributions to the dissertation

I accept the contributions formulated by the PhD student, which are summarized in the section "Contributions of the dissertation" and can be systematized as scientific, applied and applied as follows:

Scientific contributions:

1. It has been confirmed that the application of DCTs leads to dispersion, more uniform distribution and increase in the number of secondary phases separated from the solid solution, as well as to an increase in the density of dislocations, which is a major factor in improving the strength characteristics of aluminum alloys.
2. It has been proven that the sequence of heat treatment application on aluminum alloys (quenching + artificial aging) and DCT has a significant impact on the result, with the most favorable characteristics being achieved when performing DCT after aging, in contrast to conventional schemes in which DCT is applied after quenching, before the aging process.
3. It has been confirmed that the DCT does not impair the corrosion resistance of aluminum alloys, but even leads to a slight improvement, which is important when operating in aggressive environments.

Scientific and applied contributions:

1. The specific reaction of different groups of thermally hardened aluminum alloys to DCTs has been established, and for casting alloys of the AlSi7Mg type, an increase in strength without loss of ductility is reported, while in the case of deformable alloys of the AlSiMgMn system, the effects are mainly aimed at improving plasticity while maintaining strength and rigidity.
2. It has also been found that in thermally non-hardenable deformable alloys of the Al-Mg system (AlMg6, AlMg4,5), the effect of DCT is more pronounced after hardening by

plastic deformation than in the annealed state.

3. An innovative, environmentally friendly and economically justified technological process has been developed for strengthening thermally non-reinforcing and for enhancing the aging effect of thermally reinforcing aluminum alloys through the application of DCTs.

4. The applicability of DCT as a final treatment has been proven, given that it does not cause an increase in the roughness of surfaces superficially reinforced by diamond smoothing aluminum alloys.

5. Regression models have been created, which describe the relationship between the parameters of the SCO and the obtained mechanical and operational characteristics of the thermally hardened and thermally non-hardenable aluminum alloys. The models allow predicting expected results and support the selection of optimal technological modes by quantifying the influencing factors.

Applied contributions:

1. Practical guidelines have been developed for the selection of technological regime for reinforcement with DCTs, which allow for a wide range of changes in the properties of thermally reinforcing and thermally non-reinforcing aluminum alloys, according to the required performance characteristics.

2. A technological process for hardening by means of DCT, with a duration of 24 hours, has been developed, which leads to an additional increase in the strength mechanical characteristics of thermally and mechanically strengthened aluminum alloys, as well as of aluminum alloys in annealing state, while not reducing the ductility. The process is suitable for implementation in industrial conditions, where an increase in strength is required without deterioration of the plasticity of the material.

3. A technological process for hardening by means of DCT, lasting 48 hours, has been developed, which increases the ductility of thermally and mechanically strengthened aluminum alloys, as well as aluminum alloys in annealing state, while slightly increasing the strength mechanical characteristics. The process is particularly suitable for applications where a combination of improved ductility and sufficient strength is required, such as in the transport and aviation industries.

The research results achieved are relevant and significant in scientific and scientifically applied aspect and can be successfully used for future scientific and practical developments.

6. Assessment of the personal involvement degree of the PhD student in the contributions

I believe that the PhD student has personally participated in the various stages of research in the dissertation to a significant extent. The presented scientific, applied and applied contributions are the personal work of Mag. Eng. Plamen Stoyanov, under the scientific and methodological guidance of his scientific supervisors Assoc. Prof. Daniela Spasova and Assoc. Prof. Diyan Dimitrov. Proof of this are the publications of the PhD student and the participation in two research projects during his training period.

7. Evaluation of the publications related to the dissertation

The results of the dissertation research obtained by the author have been published in 4 scientific articles. All four articles have been reported at international scientific conferences held in Bulgaria and Greece, one of which is indexed in the Scopus database. One publication has not yet been published but has been accepted for publication by the publisher. In one of the articles, the PhD student is a single author. The publications are made in the period 2024-2025 and cover the topics of the presented dissertation, reflecting the main results and contributions achieved.

No data has been provided for citations found up to the time of publication of the dissertation. The PhD student has also participated in 2 scientific projects with topics related to the

topic of the doctoral thesis.

I assess the publication activity of the PhD student as sufficient, and I believe that the results of the work on the dissertation have become sufficiently known to the Bulgarian and international scientific community.

The publications on the dissertation in quantitative and qualitative terms fully satisfy the requirements for acquiring the educational and scientific degree of "Doctor".

8. Application of the results obtained

No evidence (such as business innovation certificates and/or patent certificates) for the practical implementation of the results of the research and development in the dissertation are presented. Nevertheless, I believe that the research in the dissertation and the results achieved can be successfully used both as a basis for further research purposes and tasks related to the application of the CAT method for other types of materials, as well as for solving practical problems related to the achievement of specific performance characteristics of the aluminum alloys studied.

9. Opinions, remarks and recommendations

The dissertation is formatted in accordance with the requirements of the Regulations for the Implementation of the Law and the relevant Regulations of the Technical University of Varna.

I consider all the remarks and recommendations from my preliminary review of the dissertation's first version to have been considered by the doctoral student and implemented as corrections and additions to its revised version.

My recommendations to the author of the dissertation are as follows:

1. To continue with the research on the topic of his dissertation, directing them to the development and implementation of methods for practical application of SCD and diamond EDP to increase the quality and operational characteristics of real parts made of aluminum alloys.
2. To redirect the publication of the results of its future research in international journals indexed to the world-renowned SCOPUS and/or WoS databases, and especially in journals falling into a higher quartile of impact in the relevant scientific field.

Conclusion

The dissertation meets the requirements of the Law on the Development of the Academic Staff in the Republic of Bulgaria, the Regulations for the Implementation of the Law and the relevant Regulations of the Technical University of Varna. The scientific research conducted is in a current field and deals with issues that are of increased research interest. The results obtained contain scientific, applied and applied contributions, which have been made largely public and made available to the interested scientific community through their reporting to an international audience and their presentation in scientific conferences and journals.

The results achieved in the dissertation give me grounds to propose to the members of the scientific jury to **acquire an educational and scientific degree "Doctor" to Mag. Eng. Plamen Ivanov Stoyanov** in the field of higher education 5. "Technical Sciences", professional field 5.1 "Mechanical Engineering" and doctoral program "Technology of machine-building materials".

05.01.2026 Prepared by:
Varna /Prof. Dr. Eng. Stoyan Slavov/