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REVIEW

PhD thesis of MSc. Eng. Tsegaye Shume Lemmi

entitled: "Influence of vulcanization process parameters on the physico-mechanical

properties of textile materials used for the reinforcement of rubber goods" -

commissioned by the Council of Disciplines of Materials Engineering of the Lodz University

of Technology of May 12, 2023.

Technical progress forces producers of rubber products to improve constantly their manufacturing technology. Conveyor belts are complex multi-layer composites containing rubber, textiles and a steel insert, where the percentage of individual components of the belt depends on its construction and application. These components are characterized by diametrically different structural and material properties, and the resultant of these properties determines the properties of conveyor belts, including their tensile strength, corrosion resistance and fatigue strength.

Currently, in many research centers, as well as in-house research laboratories, intensive work is being carried out on the possibility of improving the operational properties of rubber products, which include conveyor belts. The research topic undertaken by the PhD student is fully justified. The reviewed doctoral thesis is part of the discipline of materials engineering. The results of the considerations and experiments carried out by the author are a valuable source of information for practical applications in the industry in the design and implementation of new technologies for the production of rubber products. The research

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contained in the work also enriches science with new information on the problems raised by the author, which have so far been presented in the technical literature, usually in a fragmentary way.

The work containing 115 pages of text consists - excluding the author's declaration, abstract, acknowledgment, list of papers, conferences, figures, tables and acronyms - of the following chapters: Introduction, Literature review, Materials, Methodology, Experimental tests, results and discussion, Conclusions and recommendations. The work ends with a bibliography containing 135 items. A significant number of publications on the analyzed issues also confirms the relevance of the research topic undertaken by the PhD student. The bibliography mainly consists of the latest items of world literature.

In the introduction, the author justifies the desirability of taking up the topic of the dissertation and describes the main challenges faced by conveyor belts. Then MSc. Tsegaye Shume Lemmi presents the purpose of the work, description of the problem, justification for the choice of research, hypothesis and structure of the dissertation.

The main goal of the work was defined as "The main goal of this thesis work was to investigate the influence of vulcanization process parameters on the physio-mechanical properties of textile materials utilized for the reinforcement of mechanical rubber products, mainly for conveyor belt reinforcement". The thesis of the work is the statement that "The thermal aging parameters cause a physio-mechanical deterioration of textile fabric-reinforced conveyor belts. However, it is feasible to optimize the vulcanization process parameters of the textile-reinforced conveyor belts to enhance the mechanical and physical properties of the conveyor belt." Hypothesis was divided into three points: Thermal aging of synthetic yarn and fabric causes mechanical property deterioration, The vulcanization process has a negative impact on the reinforcing materials, It is possible to optimize the property of conveyor belts carcass and processing parameters. Such a thesis is fully justified in the context of the conducted research.

In my opinion, the layout of the first chapter is unusual and illegible. Typically, the introduction includes basic introductory information that brings the reader closer to the subject of the doctoral dissertation, while theses are placed in subsequent chapters of the work. The correct order of the chapters: 1 - introduction, 2 - literature review, 3 - purpose, thesis and scope of research.

The literature review in Chapter 2 consists of as many as eleven subchapters. In my opinion, it could be divided into a maximum of 3-4 subsections. In the first part, the author lists mechanical rubber products, gives the range of vulcanization times and temperatures, and describes the development of rubber products in historical terms. In the second and third subchapters, the author discusses in detail the construction of belt conveyors, gives the differences in the construction of tapes in terms of construction, properties and their purpose. However, in subsections 4-10 he discussed: properties and use of textiles for reinforcing conveyor belts, quality requirements for belts, areas of their application, vulcanization parameters. The literature review ends with a summary, in which the PhD student justifies the main directions and the need to conduct research in the analyzed topic of the dissertation. Apart from the fact that the review is broken down into subsections in too much detail, I can say that the review was carried out correctly, indicating the trends of currently conducted scientific research and implementation works on the technology of manufacturing rubber products. At this point, I would like to emphasize a very high level of literature review, which proves a good level of preparation of the candidate for writing a doctoral dissertation.

In the third chapter - materials, the properties of polyester yarns, industrial fabrics and styrene-butadiene rubber are characterized. In the next part of the work, four subchapters discuss the research methodology: experimental design from yarn to conveyor belt, thermal aging of high tenacity polyester yarn, thermal aging of woven fabric and modeling and vulcanization parameters of textile-reinforced conveyor belts. Despite the efforts of the PhD student, this chapter contains some inaccuracies, e.g. page 41 "...Prior to the thermal aging

of each high tenacity polyester yarn sample, the industrial oven was heated above the designated temperature for the thermal aging to avoid the possibility of temperature drop while placing the yarn samples in the oven". Right approach, but by how many degrees did the oven temperature rise?

Then, in Chapter 5, experimental research was presented, which consisted of subsections: Experimental investigations of high tenacity polyester yarn, Tensile property experimental test of industrial woven fabric samples and textile reinforced conveyor belts. In my opinion, the name of chapter 5 - Experimental tests, is misleading because it suggests the results of research. This chapter is a continuation of chapter 4 - Methodology. There is no table of test results in Chapter 5. Chapters 4 and 5 should be combined into one and the results of the research should be added in the tables, the correct title of the chapter should be: methodology and research results, and then in the next chapter, the obtained results should be discussed using graphs and photos.

The most valuable part of the reviewed doctoral thesis is chapter 6 - results and discussion. In section 6.1 the experimental test results of high tenacity polyester yarns were presented, and the findings are thoroughly discussed from the perspective of the usage of the material in the conveyor belt application. As part of the work, based on TGA thermogravimetric tests and DSC scanning diffraction calorimetry, an analysis of thermal properties of polyester yarn was carried out. This is the right approach given that these materials are subjected to heat treatment during rubber vulcanization. Based on the TGA tests, the author determined the distribution and thermal stability of polyterephthalate yarn. The results obtained from the Thermogravimetric Analysis (TGA) and Diffraction Scanning Calorimetry were significant in determining the optimum vulcanization temperature for the conveyor belts reinforced by fabrics made from polyester yarns in the following sections of the work. The next stage of the work was the analysis of strength properties of high-strength polyester yarn. It has been shown that the temperature and aging time have a significant effect on the yarn properties, especially on its elongation to

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break. Next, the work presents Influence of Thermal Aging Parameters on the Tenacity of Industrial Polyester Yarn. These studies contain valuable information on the effect of temperature on the strength of polyester, not only from a scientific but also from an industrial point of view. It has been shown that the tenacity of the yarn samples remains the same if the yarn undergoes thermal aging below its glass transition temperature. The aging of yarn above the glass transition temperature of the yarn gradually deteriorates the yarn's tenacity, mainly if the aging temperature is above 220°C, the tenacity loss is higher. Studies of plastic properties have shown that at high temperatures, exceeding 200°C, the elongation of PET yarn increases very quickly, at 220°C by almost 300%. According to the author, the thermal aging of polyester fiber or yarn can cause the formation of irregular breakage, coiling, or necking on fiber breakage under the tensile load.

In chapter 6.2, the PhD student focused on the analysis of EP Woven Fabrics Experimental Test Results. In this part of the work, the author successively analyzes the influence of Resorcinol-Formaldehyde-Latex (RFL) adhesive solution on the tensile property of EP woven; Effect of Thermal Aging on the Tensile Strength of EP Woven Fabric; Effect of Thermal Aging on the Percentage Elongation of EP Woven fabric. In the summary of this part of the work, the PhD student stated that the fabric used to reinforce the conveyor belt is expected to have a lower elongation at break while having high tensile strength. Therefore, the vulcanization parameters during conveyor belt production must be defined based on the fiber composition of the fabric carcass and appropriately controlled to achieve these requirements.

Chapter 6.3. The work entitled Analysis of EP Woven Fabric Reinforced Conveyor Belts is an extremely valuable source of information about the influence of belt vulcanization parameters on their properties. These studies have a very large application potential and will undoubtedly be used in the industry in the implementation of new technologies for the production of rubber products. In this part of the work, among others, the influence of vulcanization parameters on the properties of individual components of the conveyor belt

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was analysed. The effect of vulcanization on the strength properties and elongation of conveyor belts was determined. The study was supplemented by the analysis of the effect of Vulcanization Parameters on the Adhesion Strength Between Plies of the Textile Reinforced Conveyor Belts.

The last chapter of the work consists of conclusions and recommendations. In this chapter, in a synthetic way, MSc. Eng. Tsegaye Shume Lemmi presented and confirmed the novelty of the obtained research results and their cognitive nature. According to PhD student based on the thermal aging and vulcanization parameters considered in this work and experimental investigations conducted on the yarns, fabrics, and conveyor belts, the vulcanization parameter of 160°C for 35 minutes was found to be an optimum parameter to vulcanize three layers of the textile-reinforced conveyor belt. The obtained research results unequivocally confirm the achievement of the ambitious goal and prove the thesis of the work.

Work Notes:

Doctoral dissertation of MSc. Eng. Tsegaye Shume Lemmi, in addition to the above-mentioned advantages, also has fragments that require a broader discussion. Substantive comments are presented below:

1) page 33 - what did the Author mean when he wrote: "......The woven fabric samples were supplied in a greige and dipped form...". I assume what the author meant, please clarify.

2) Page 40 – Please comment "..The aging duration of six minutes was added to the experiment only for the yarn samples that underwent thermal aging at 220°C..", why 6 minutes at 220°C.

3) Page 41 - table 4. How the variants were selected, for the reader it looks as if the variants were selected at random. For example: linear densities - 990 tex, for 35 minutes, aging only at 160°C.

4) Page 48 – Section 5.1.1 "..For the DSC experimental tests, polyethylene terephthalate yarn samples of 5-10 milligram weight were folded in an aluminum pan. This runs were conducted at running a temperature up to 360°C while heating 10°C/min...". Heating rate different than vulcanization. Does the PhD student believe that the rate of heating affects the obtained TGA results?

5) Pages 66 and 67- Figures 42 and 43 show error bars on the graphs. With similar tenacity values, large differences in error sizes were obtained for individual variants. It follows from this that the time and temperature of aging affect the size of the dispersion of results? please comment. Excessive differences in the size of errors for individual variants are also present in the following figures.

6) Page 70 - in Fig. 46, the curves representing the "dipped" and "Greige" variants have the same colors. Too many variants on the figure.

7) Pages 75 and 76 - Figures 49 and 50 show very interesting pictures of the surface of the fibers. At what time and temperature were the first cracks recorded? In the opinion of the PhD student, did these microcracks and surface damage affect the strength properties of the fibres?

8) Sections 6.2.2 and 6.2.3 describe the effect of aging on the strength and ductility properties of EP woven fabric. Comparing the results presented in Figures 52 and 54 as well as 53 and 55, it can be seen that the aging temperature of the fabrics has a much greater influence on the plastic properties than on the strength properties. Why such big differences. Typically, an increase in plastic properties causes a decrease in plastic properties. Please comment.

9) Page 52 - Figure 30. Fabric specimen dimension for tensile test. Marking of the sample inconsistent with the rules of dimensioning. Which means 50, 100 and 220 mm; thickness, width, length?

10) Page 54 – "..The dimensions of the conveyor belt specimens were prepared according to the norm, as shown in Figure 32...". There are no sample sizes in the drawing.

11) Page 54 - "..The experimental test was conducted in standard laboratory conditions and a machine testing speed of 100mm/min...". Have tests at different draw speeds been considered? 100 mm/min is very slow. During operation, belts are subjected to dynamic loads.

12) Page 61 - The author stated that ".the increase in the linear density of the yarn has no significant change in the tenacity of the yarn (± 1 cN/tex), as shown in Figure 40...." Indeed, the value of the total elongation to break for the analyzed variants is similar. However, at an elongation of 12% (about 80% of the total elongation), the differences in strength are about 15%. Please comment.

13) Page 62 - illegible Figure 41. General overview of tensile property behavior of 110 tex-basic yarn under various thermal aging conditions. Too many lines and colours, the reader can get confused about what is what. This drawing should be made in the xy system; temperature on the x-axis, tenacity on the y-axis. Alternatively, in the x,y,z system: time, temperature, tenacity, e.g. in Statistica program. This would make it possible to determine the approximating function of two variables.

Final conclusions:

Taking into account the timeliness of the selection of the topic, which is of very significant cognitive and practical importance, the correct and valuable hypotheses of the dissertation, which has been fully proven, as well as the skills of the PhD student who:
- showed very good mastery of the scientific workshop in the discipline of materials engineering and very extensive practical experience in the field of testing fiber materials used to reinforce rubber products
- was able to apply many modern research methods, e.g. research on the effect of glue impregnation of fabrics on its adhesion to rubber, research on the impact of aging time and temperature on the physicochemical properties of yarns, fabrics and conveyor belts. He

showed unequivocally that the appropriate selection of parameters of the vulcanization process makes it possible to improve the operational properties of conveyor belts

- formulated correct and valuable conclusions resulting from the results of a very wide range of experiments, which, apart from high cognitive values, are characterized by high application potential. This is confirmed by the fact that this work was carried out in cooperation with Sempertrans Belchatów, the world's leading manufacturer of conveyor belts.

The dissertation of Mr. Tsegaye Shume Lemmi makes a significant contribution to the development of the scientific discipline of materials engineering at the same time meets the formal requirements for dissertations for the degree of doctor, in accordance with the regulations on scientific degrees and titles contained in the Law of July 20 , 2018. Art.187 pt.1 and pt. 2 "Law on Higher Education and Science". I hereby request the Council of Disciplines of Materials Engineering of the Lodz University of Technology of to admit the Author to further stages of the doctoral dissertation.

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