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Use of Thermomechanical Analysis in the Design of a Composite System with a Low Coefficient of Longitudinal Thermal Expansion for End Gauges

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The work presents the possibilities of using the thermomechanical analysis to design composite systems with carbon fibers, which should have a low coefficient of longitudinal thermal expansion will be potentially valuable in the form of calibrated length gauges. ChS Epoxy 520 epoxy resin (hardener T0492) was used as a matrix for composite production. Carbon fibers were tested in the form of short recycled fibers and continuous fibers in roving and fabric. The values of the coefficient of longitudinal thermal expansion of all prepared systems were determined below the glass transition temperature of the polymer matrix in the temperature range of 5º to 40ºC, i.e., in the range suitable for use in technical practice. From the obtained results, it is evident that carbon fibers significantly influence the epoxy matrix's behavior. The coefficients of longitudinal thermal expansion of the epoxy resin in this temperature range from 47.6 to 65.2 [10^{-6}/K], the coefficients of the composite system with recycled carbon fibers with a random arrangement from 38.6 to 47.6 [10^{-6}/K], coefficients of systems with a parallel array of fibers from to 3.4 to 2.7 [10^{-6}/K] for carbon fibers in the form of roving and 4.3 to 2.0 [10^{-6}/K] for carbon fibers in fabric form.

Keywords: Thermomechanical Analysis, Carbon Fibers, Epoxy Resin, Coefficient of Longitudinal Thermal Expansion

1 Introduction

End gauges are the most accurate material standard for measuring length in technical practice. Their standard composition often limits engineering companies that use end gauges during production. Most commonly used gauges are made of steel and are therefore not suitable for corrosive environments, such as those found in galvanizing plants. Alternatively, ceramic gauges which do not have undesired electrical conductivity or gauges made of carbide can be used. The measurement accuracy is fundamentally affected by the magnitude of the longitudinal thermal expansion of the material from which the end gauge makes, which according to EN ISO 3650, is stated with an uncertainty of ± 1 micrometer per meter temperature changes by 1 ° C. The values of longitudinal thermal expansion of standardly used end gauges for the KM set for micrometer gauges in the range of 2.5 to 25 mm in accuracy classes 0 and K are as follows: steel 11.5 [10^{-6}/K], ceramic 9.5 [10^{-6}/K] and carbide 4.5 [10^{-6}/K]. The leading manufacturers of end gauges today are Mitutoyo, Mahr, Koba, and Insize. The Insize steel set price is around 7 thousand CZK; ceramic sets Insize is 13 thous. CZK. The Mitutoyo steel set cost is approximately 16 thous. CZK, Mitutoyo ceramic sets are 29 thous. CZK. The price of the Mahr steel set is around 36 thous. CZK, ceramic sets Mahr is 43 thous. CZK. The price of the Koba steel set is about 50 thous. CZK, ceramic sets Koba is 56 thous. CZK. Polymer end gauges are not commonly used due to the high thermal expansion of all polymeric materials, which is significantly higher than steel. For example, unfilled epoxy resins show a longitudinal thermal expansion of 55 to 65 [10^{-6}/K], and thermoplastics offer values more than double. According to the current survey, using composite materials as materials for end gauges is not addressed at the national or international level. [1, 2, 3, 4] Composite materials have been used advantageously for many decades in some industries. Over time, carbon fibers in various forms have become the most effective reinforcing material for polymer matrices. In addition to the generally known high values of mechanical parameters, carbon fibers also have a low coefficient of longitudinal thermal expansion in the range from -1 to 8 [10^{-6}/K]. This property was used to design the composition and structure of composite samples. The content of the presented work compares the effect of short recycled carbon fibers and continuous carbon fibers stored in an epoxy matrix on the reduction of the coefficient of longitudinal thermal expansion in the laboratory process of sample preparation. [4, 5, 6]

2 Materials and Methods

Two-component low molecular weight epoxy resin ChS-EPOXY 520 (Districhem a. S.). was chosen as a

matrix for the production of composite samples. Hardener T0492, added in a weight ratio of 100:27 (Districhem a. S.), Was used for curing. The coefficient of longitudinal thermal expansion of epoxy resins is in the range of 45 - 55 [10^{-6}/K] depending on the temperature. Carbon fibers were used in three forms. CarbisoTM MF short recycled fibers (Easy Composites Ltd.), carbon roving 3700 tex 50K. (HAVEL COMPOSITES CZ, s.r.o.) and carbon fabric 800 tex 12k (HAVEL COMPOSITES, CZ, s.r.o.). The fabric is interwoven with 7.6 tex polyester fibers perpendicular to the longitudinal arrangement of the carbon fibers. The specific weight of the material was 380 g/m². The coefficient of longitudinal thermal expansion of carbon fibers ranges from -1 to 8 [10^{-6}/K] this range includes the coefficient of thermal expansion of fibers in the longitudinal and transverse direction. A thermomechanical analyzer model TMA PT-1000LT was used to measure the coefficient of longitudinal thermal expansion. The instrument is used to measure physical quantities of solids (plastic, metal, glass, ceramics, etc.) as a function of temperature and time in the temperature range from -150°C to 1000°C heating rate 0.1 to 50°C/min. The longitudinal thermal expansion coefficient was measured in samples for the temperature range 5°- 40°C, which is the most important from everyday technical use. The interphase quality was evaluated from images of fracture surfaces of prepared systems using a TESCAN MIRA3 scanning electron microscope. Composites samples fracture surfaces were sputtered with a Pt-Pd layer (Quorum Q150R ES) with a thickness of 2 - 4 nm.

3 Experimental

The samples were prepared in two ways according to the type of carbon fibers used - *Fig.1*. Samples with recycled carbon fibers were prepared by casting a mixture of epoxy resin - short carbon fibers into a silicone mold - *Fig. 2*. The sample preparation mold was made to meet the size and shape requirements for measuring the coefficient of longitudinal thermal expansion using a thermomechanical analyzer TMA PT-1000LT. Samples of epoxy resin with continuous carbon fibers were prepared by layering a roving or fabric into a silicone mold - *Fig. 2*. Curing of all types of samples took place for 24 hours at room temperature 22 ± 2ºC and overpressure 0.7 ± 0.1 MPa in a pressure vessel. After removing the mold, the pieces were cured at 60 ± 2°C for 10 hours. By using overpressure, cavities and bubbles in the volume of all prepared composite samples were minimized. Pieces of roving and fabric were adjusted to the required dimensions according to the measuring device's needs. The samples' composition and structure were designed to make the most of the presence of carbon fibers. In recycled carbon fibers, the fiber filling was 40 phr, which is the highest possible level of fiber content that is economical and technologically feasible. [7]

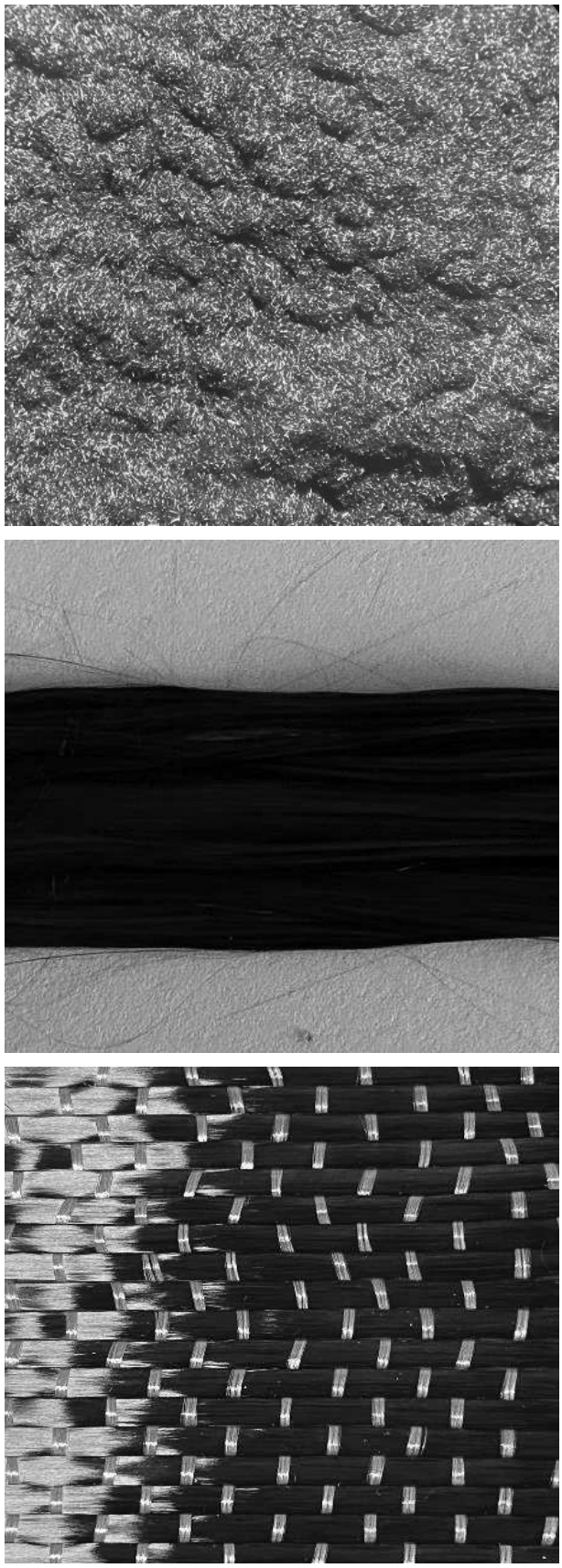


Fig. 1 *Images of Used Carbon Fiber Materials - Recycled Fibers - Roving - Fabric*

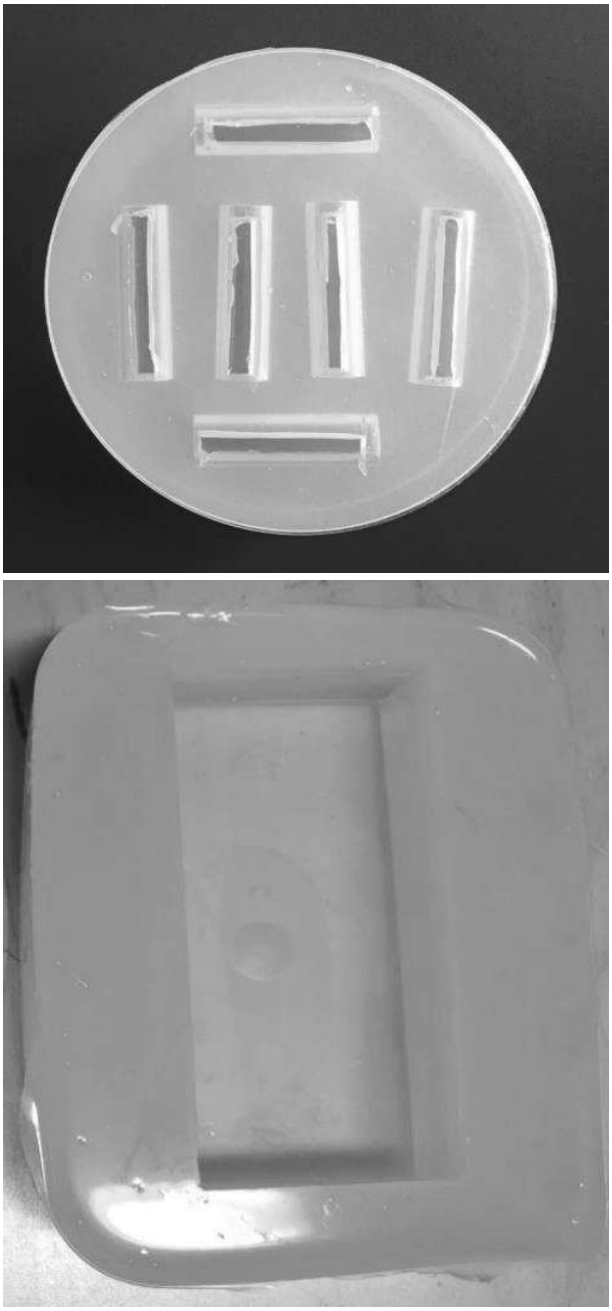


Fig. 2 *Images of Silicone Molds for Sample Production - for Casting Technology and for Layering Technology*

In the roving and fabric case, the weight ratio of fibers to the matrix was designed to be 1:1 so that all fibers in the proposed system were perfectly wetted and samples could be prepared under laboratory conditions. To measure the longitudinal thermal expansion coefficient, a thermomechanical analyzer model TMA PT-1000LT was used - *Fig. 3*. The samples and the standard were first cooled from room temperature to 5°C, where they were tempered for 5 minutes. They were then heated to 40°C at a rate of 0.5°C per minute. Tempering was not performed at each of the selected temperatures because for samples of given dimensions, the omission of tempering does not significantly affect the measurement results. The measurement conditions were set - temperature rise, temperature to which the measured material is heated or cooled, and endurance at this temperature. The heating took place in an electric furnace, with which the device is equipped. Cooling was performed with liquid nitrogen. The measurement results were evaluated in the Evaluation program, in which the measured temperature-dependent dilatation curve was read together with the correction curve. The program automatically corrected the dilatation curve. The curve of the coefficient of thermal expansion as a function of temperature was subsequently generated from the plotted curve. From this curve, the coefficient of longitudinal thermal expansion was evaluated using the process ALPHA_PHYS.

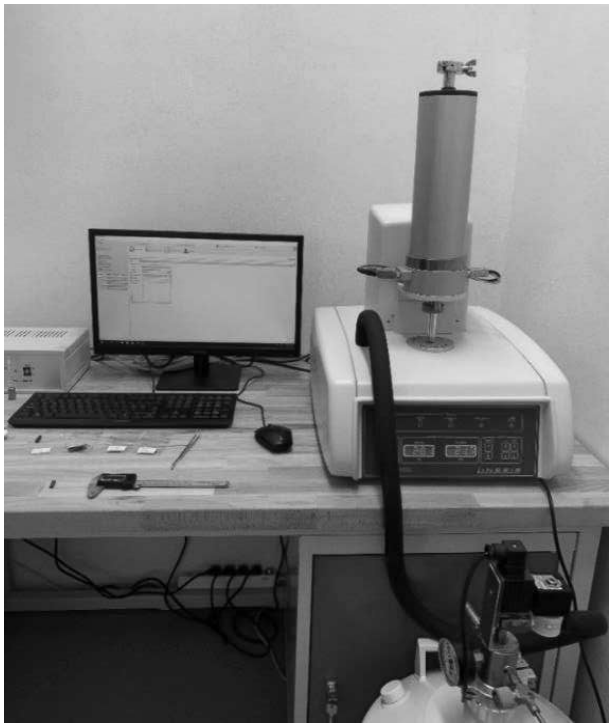


Fig. 3 *An Image of Thermomechanical Analyzer Model TMA PT-1000LT*

4 Results

Four groups of samples differing in composition and structure were prepared. The first group of samples consisted of samples of pure epoxy resin. The second group consisted of samples prepared by mixing 40 sheets of recycled carbon fibers with epoxy resin. Carbon fibers with a diameter of 8 micrometers and a length of 100 micrometers were randomly arranged in the samples. The third group consisted of samples formed from carbon roving embedded in an epoxy matrix. These strands were arranged in parallel, impregnated with epoxy resin, and layered in a silicone mold. The fourth group of samples was prepared by impregnating the carbon fiber fabric with epoxy resin and laminating it to a silicone mold. The ratio of carbon fiber to epoxy resin was 1:1 in both types of

samples. For each material composition, ten samples were prepared, which were machined and then measured. The coefficient of longitudinal thermal expansion α was evaluated at temperatures of 5º, 10º, 15º, 20º, 25º, 30º, 35º, and 40°C. The temperature rise in the measurement was set at 0.5°C/min, the endurance at the start and end temperature was 5 minutes. In the Evaluation program, the sample's measured dilation curve as a function of temperature was read together with the correction curve, according to which the program automatically corrected the dilation curve. From this curve, the coefficient of longitudinal thermal expansion was evaluated using the function ALPHA_PHYS. Subsequently, the curve of the coefficient of thermal expansion depending on the temperature was generated - *Fig. 4*. The arithmetic mean of all measured samples of the given type was calculated from the measured values, and the measurement uncertainty for individual temperatures was determined. The calculated uncertainty following the document JCGM 100:2008, and its calculation was performed using an algorithm borrowed from CMI OI Liberec. The resulting coefficients of longitudinal thermal expansion for individual types of samples and temperatures are given in the appendix to *Tab. 1* and graphically displayed - *Fig. 5*.

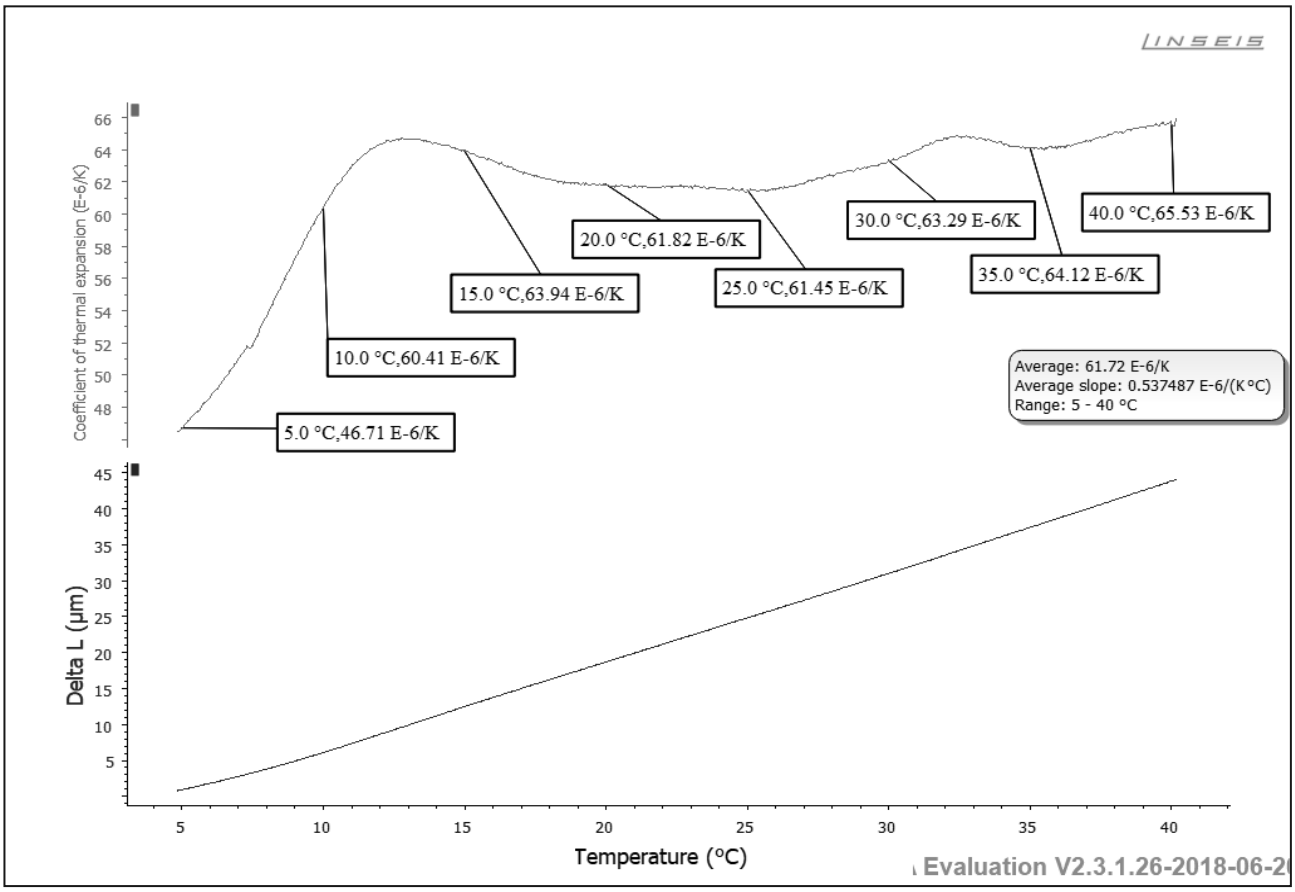


Fig. 4 *Dependence of the Coefficient of Longitudinal Thermal Expansion and Expansion on Temperature for a Sample of Pure Epoxy Resins*

Tab. 1 *Average Values of Coefficients of Longitudinal Thermal Expansion for Individual Types of Samples Depending on Temperature with Calculated Values of Measurement Uncertainty*

temperature	epoxy resin pure	epoxy resin carbon fibres recycled	epoxy resin carbon fibres roving	epoxy resin carbon fibres fabric
	average values of coefficients **α [10⁻⁶/K] /** measurement uncertainty **U [10⁻⁶/K]**			
5 °C	47.6/2.7	38.6/2.5	3.4/2.2	4.3/2.3
10 °C	56.4/2.6	44.5/2.4	3.8/2.2	3.3/2.2
15 °C	62.4/2.6	46.1/2.5	3.3/2.2	3.9/2.2
20 °C	61.2/2.7	47.7/2.5	3.1/2.1	3.8/2.1
25 °C	61.4/2.7	46.8/2.6	3.3/2.2	3.4/2.1
30 °C	62.8/2.6	46.5/2.5	3.0/2.1	3.0/2.1
35 °C	64.2/2.6	47.1/2.6	3.1/2.1	2.3/2.2
40 °C	65.2/2.7	47.6/2.5	2.7/2.1	2.0/2.0

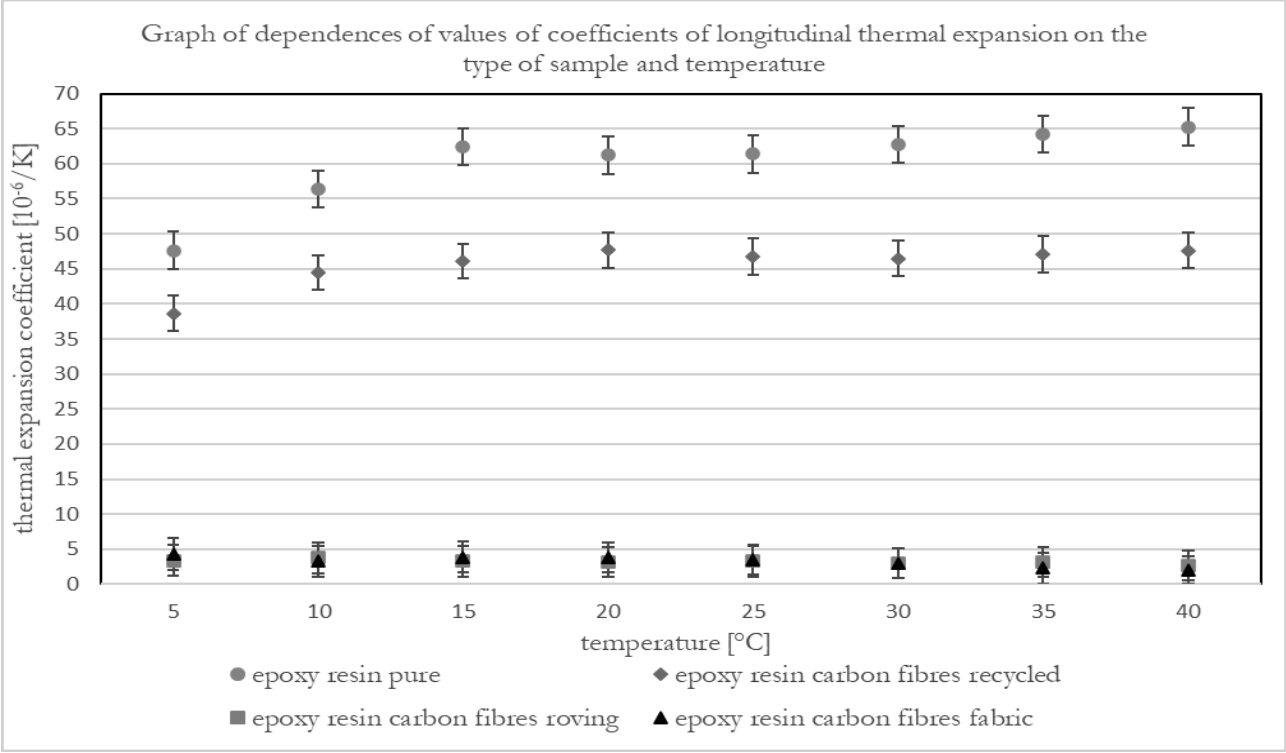


Fig. 5 *Graph of Dependences of Values of Coefficients of Longitudinal Thermal Expansion on the Type of Sample and Temperature*

From the performed and evaluated measurements of samples of pure epoxy resin and all prepared composite systems, the significant influence of their composition or presence, especially carbon fibers' arrangement, is obvious. Pure epoxy resin - ***Fig. 5*** shows a longitudinal thermal expansion coefficient in the range of 47.6 to 65.2 [10^{-6}/K] for the temperature interval 5° to 40°C. The values change significantly in the monitored interval, especially in the temperature range of 5° to 15°C, from 47.6 to 62.4 [10^{-6}/K]. In the interval 15° to 30°C, the α values practically do not change and range from 62.4 to 62.8 [10^{-6}/K]. A further increase occurs after exceeding the temperature of 30°C when the value of the coefficient α at 40°C reaches up to 65.2 [10^{-6}/K]. This increase is no longer so marked. The course of the dependence changes in different temperature intervals. First, there is a linear increase in this dependence, then a slight decrease or stagnation, and with a further rise in temperature again, there is a slight increase, which is also linear. It has to be taken into account that all expansion processes take place below the glass transition temperature of the epoxy resin. The differences in the coefficient of longitudinal thermal expansion can be attributed to the gradual release of the crosslinked epoxy resin's macromolecular structure. The composite system behaves analogously, which is made of an epoxy resin in which short recycled carbon fibers in an amount of 40 phr - ***Fig. 5.*** In the temperature range 5° to 15°C, the coefficient of longitudinal thermal expansion increases from 38.6 to 46.1[10^{-6}/K]. The further growth of the coefficient α in the temperature range of 20° to 40°C is no longer so marked. At the temperature of 40°C, it reaches 47.6 [10^{-6}/K], which is the value of the coefficient of longitudinal thermal expansion of pure epoxy resin at 5°C. A comparison of the two groups of samples shows that the effect of carbon fibers' presence is evident. At a temperature of 40°C, the filled epoxy resin shows the same longitudinal thermal expansion coefficient as pure resin at 5°C. The short randomly arranged carbon fibers in the epoxy matrix can prevent the thermally conditioned gradual release of the macromolecular structure by their presence or interactions at the interfacial interface. The fibers' effective use is not possible because they are short (average length is 100 micrometers) and randomly arranged. Due to these parameters, they can no longer be used in the system, and their effect on reducing the coefficient of longitudinal thermal expansion is limited. The increase in the epoxy resin's fiber content has virtually no further significance for this physical property. [7] Graph of the dependence of the coefficient of longitudinal thermal expansion for the system epoxy resin - recycled carbon fibers - ***Fig. 5*** practically copies the graph for pure epoxy resin. Overall, it is shifted to lower values of the coefficient of longitudinal thermal expansion. The effect of using continuous carbon fibers in the form of roving or fabric is significant. Both in the case of roving and the point of cloth, there is a marked change in the values of the coefficient of longitudinal thermal expansion - ***Fig. 5.*** In both cases, the coefficient α does not exceed the value 5.0 [10^{-}

^{6}/K] in the temperature range from 5° to 40°C. This behavior is primarily due to the presence of long carbon fibers. Their essential characteristic is a low coefficient of longitudinal thermal expansion. The parallel arrangement of the fibers has a fundamental influence, which will enable the use of this physical property in the evaluated direction. The minimized amount of epoxy resin serves primarily to join the fibers into the desired shape. It can be seen from the values given in ***Tab. 1*** that there is no significant difference in the use of carbon fibers in the form of roving or the shape of the fabric. In contrast to the two systems described above, pure epoxy resins and epoxy resins filled with short carbon fibers - ***Fig. 6***, ***Fig. 7***, in the samples with arranged carbon fibers, there is a significant decrease in the values of the coefficient of longitudinal thermal expansion with increasing temperature - ***Fig. 8, Fig. 9***. There is a dominant use of carbon fibers compared to the less significant effect of epoxy resin, which introduces into the dilating composite system the instability caused by the release of the crosslinked macromolecular structure with increasing temperature - ***Fig. 8, Fig. 9***. At the same time, in ***Fig. 8*** and ***Fig. 9***, see inequalities on the upper curve - the curve of the values of the coefficients of longitudinal thermal expansion depending on the temperature. The deviations mentioned above/inequalities occur due to the stresses arising at the composite system's interfacial interfaces due to the low dilatation of the carbon fibers and the many times higher dilatation of the epoxy resin. These phenomena are evident, although the process took place below the glass transition temperature of the resin. These deviations can be observed mainly in the course of expansion curves - ***Fig. 7, Fig. 8,*** and ***Fig. 9***, in which carbon fibers of both types are present. In both cases, where the fibers are roving and fabric, these irregularities are visible on the curve. This condition's explanation is based on the low longitudinal thermal expansion of carbon fibers, which is stable over a wide temperature range, and the high longitudinal thermal expansion of the crosslinked epoxy resin, which changes with increasing temperature. When placing fibers in the form of roving and fabric and evaluating the longitudinal thermal expansion coefficient in the longitudinal direction, there are minimal changes in the fibers, but a significant difference in the resin surrounding these fibers at the interface - interphase and surrounding epoxy resin. The result leads to imbalances, which are evident in the graphs - ***Fig. 8*** and ***Fig. 9*** as small fluctuations on the curve. In transverse thermal expansion, the fibers in the composite system will not be significantly applied. The formation of said imbalances will not occur at the interfacial interface. [8] In the case of the epoxy resin system - randomly arranged short recycled carbon fibers - ***Fig. 7***, it can be seen that the fluctuations on the curve are not so significant, which testifies to the legitimacy of this explanation. Fluctuations on the curve indicate a quality interfacial interface, which was also verified using SEM analysis - ***Fig. 10, Fig. 11***.

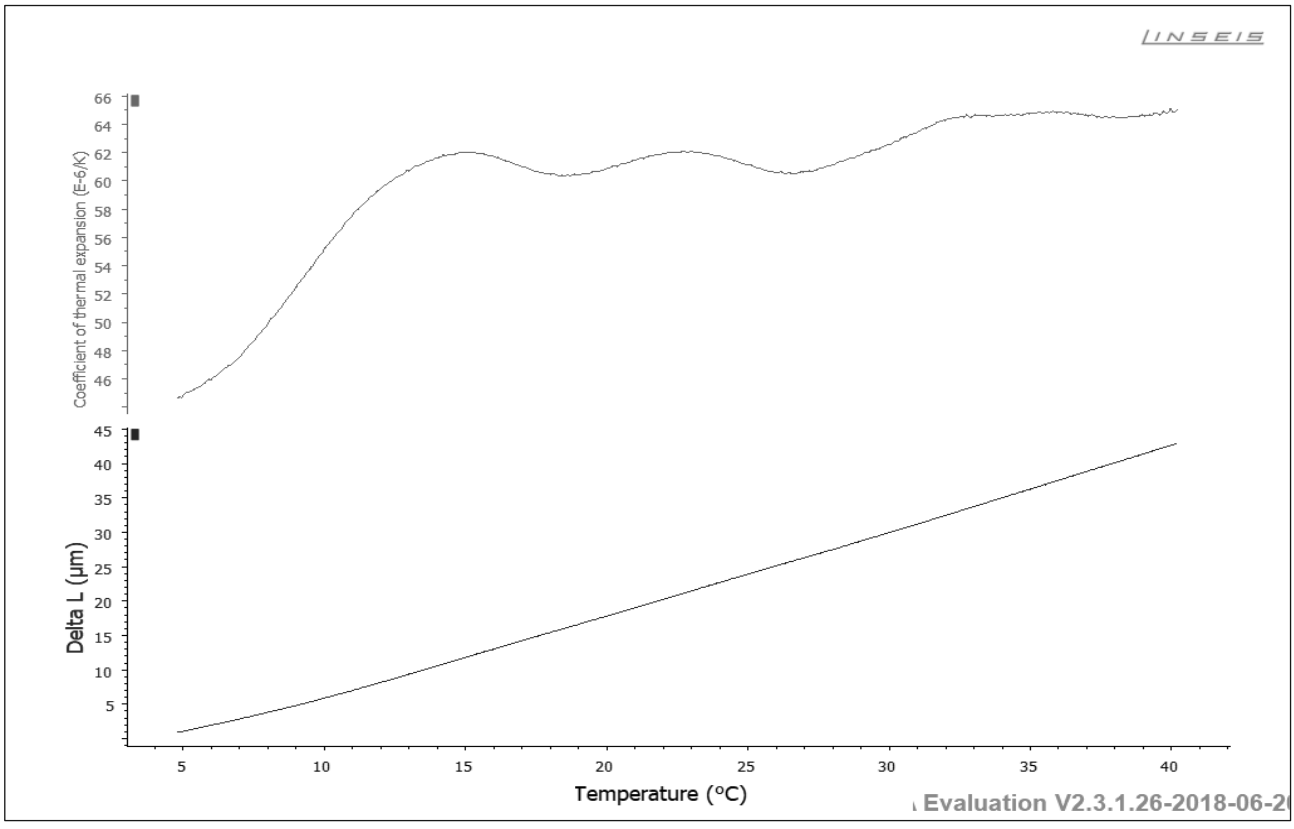


Fig. 6 *Dependence of the Coefficient of Longitudinal Thermal Expansion and Dilatation on the Temperature for a Sample of Pure Epoxy Resin*

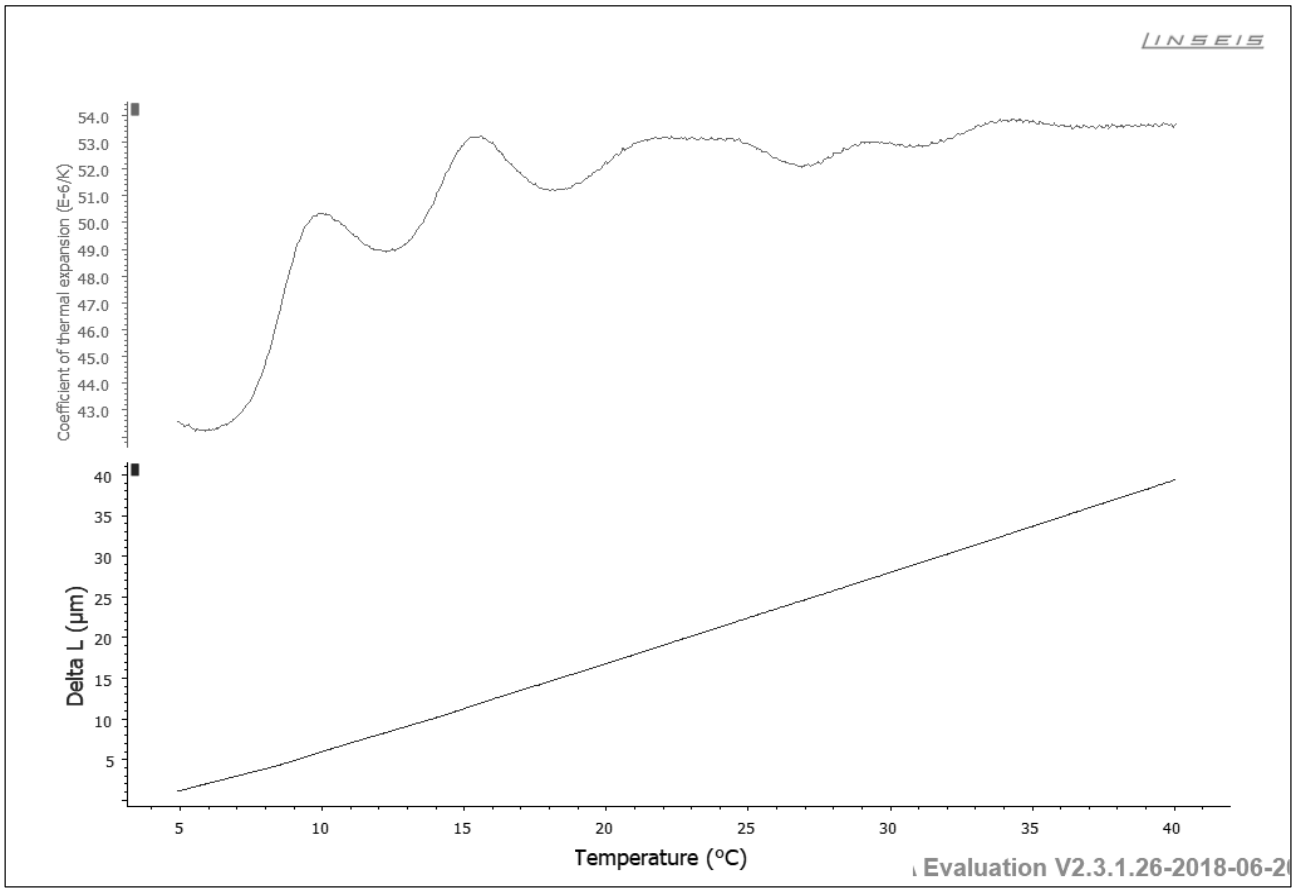


Fig. 7 *Dependence of the Coefficient of Longitudinal Thermal Expansion and Dilatation on the Temperature for a Sample of Epoxy Resin Filled with Recycled Carbon Fibers*

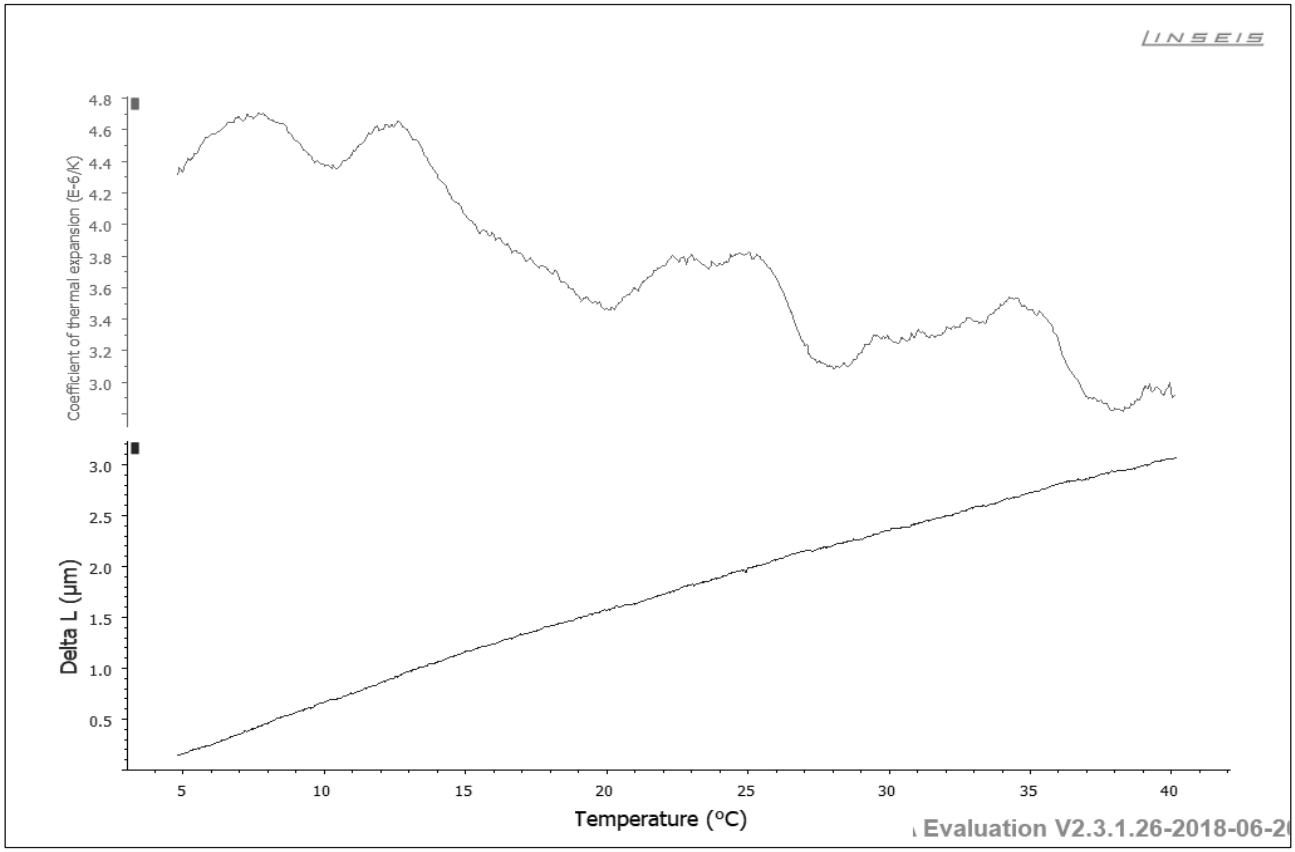


Fig. 8 *Dependence of the Coefficient of Longitudinal Thermal Expansion and Dilatation on the Temperature for a Sample of Epoxy Resin with Carbon Fibers in the Form of Roving*

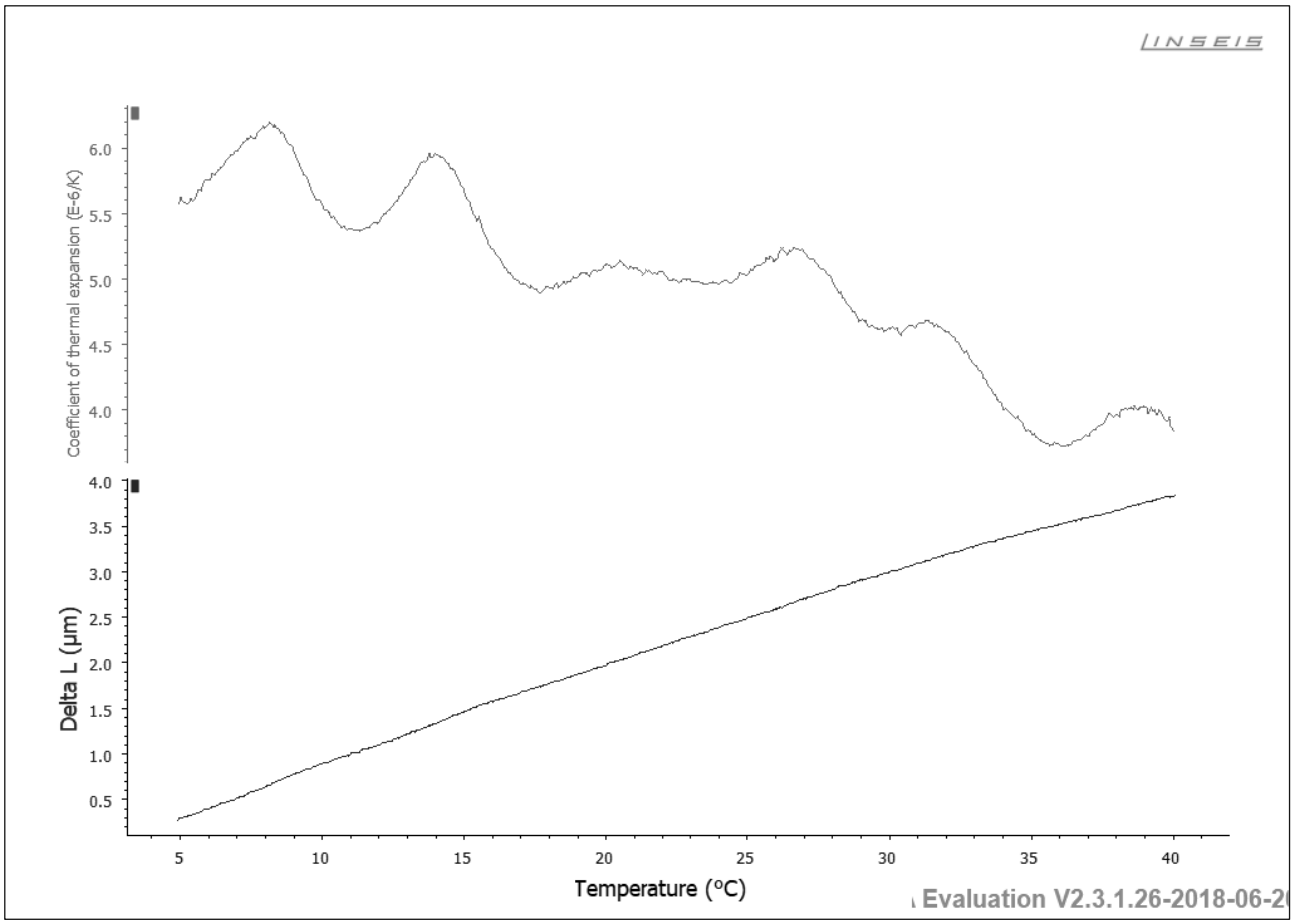
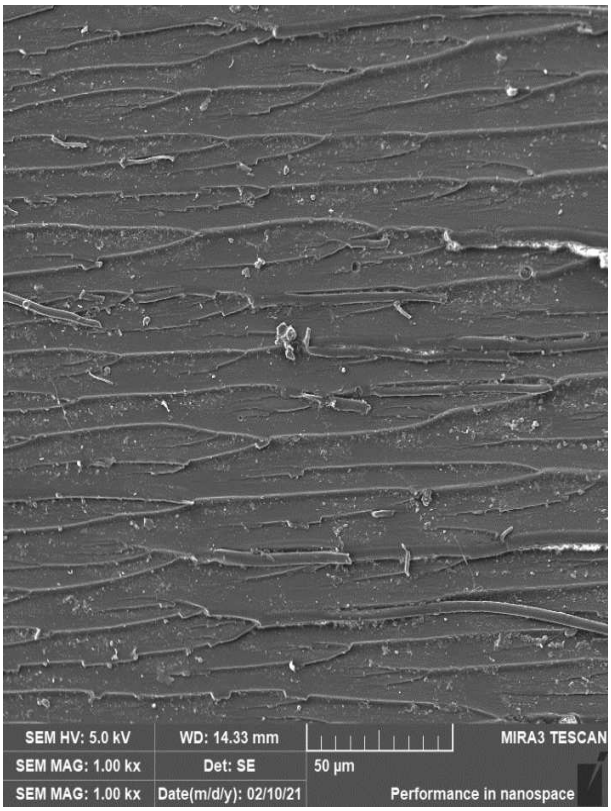


Fig. 9 *Dependence of the Coefficient of Longitudinal Thermal Expansion and Dilatation on the Temperature for a Sample of Epoxy Resin with Carbon Fibers in the Form of a Fabric*



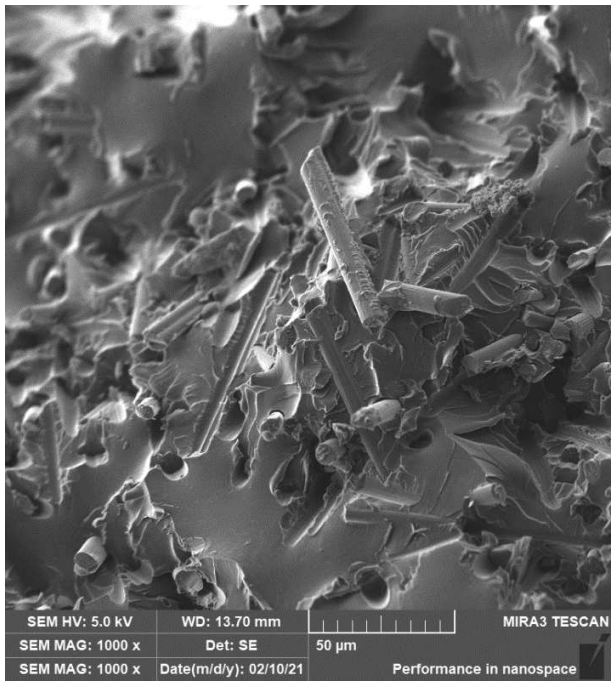
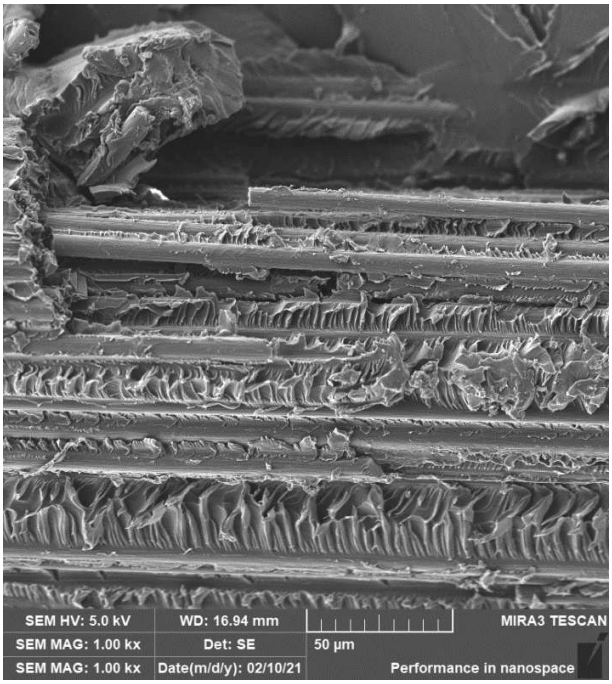


Fig. 10 *Microphotographs of Fracture Surfaces of Samples of Pure Epoxy Resin and Composite Samples with Recycled Carbon Fibers. Scanning Electron Microscope TESCAN MIRA3*



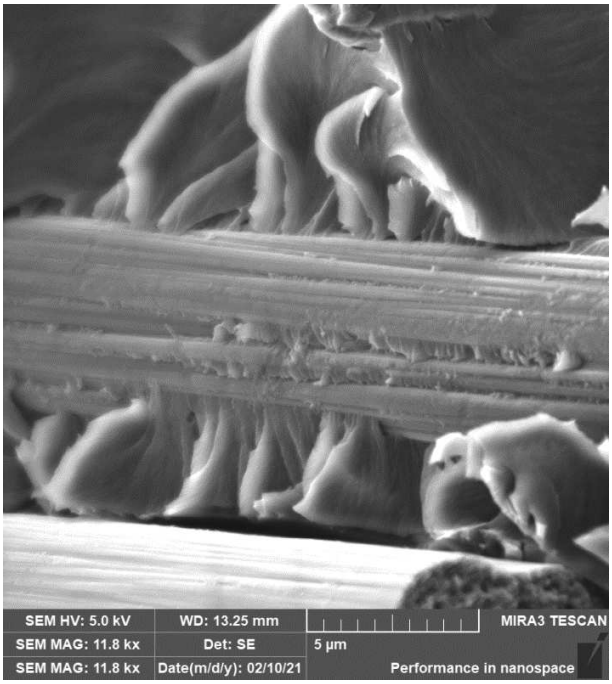


Fig. 11 *Microphotographs of Fracture Surfaces of a Sample with Continuous Fibers and a Carbon Continuous Fiber with Epoxy Resin Residues on its Surface. Scanning Electron Microscope TESCAN MIRA3*

4 Conclusion

Based on the performed experiments, it was found that by using parallel arranged carbon fibers in epoxy resin in the form of roving or fabric, the coefficient of longitudinal thermal expansion of epoxy resin can be reduced from 47.6 to 65.2 [10^{-6}/K] to values lower than 5.0 [10^{-6}/K] in the temperature range 5° to 40°C. The weight of carbon fibers to the weight of the epoxy resin was 1:1 in roving/fabric types of samples. The experiments were performed below the glass transition temperature of the epoxy resin used. The selected temperature range is adequate to the temperatures used when using these materials in standard workshop practice. The designed and verified composition and structure of the samples has the potential for use where end gauges are needed, which will meet the required parameters and at the same time will not be subject to a corrosive environment - ***Fig. 12***.

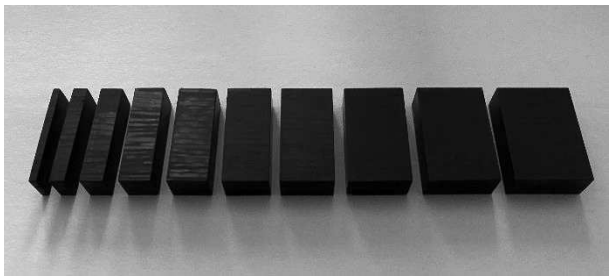


Fig. 12 *The Image of a Set of End Gauges with the Above Coefficient of Longitudinal Thermal Expansion [9]*

Acknowledgement

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