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Received (Otrzymano) 21.02.2018

PROBLEMS IN EXAMINING THERMAL PROPERTIES OF INSULATING COMPOSITE MATERIALS DESIGNED FOR TECHNICAL MEANS OF TRANSPORT

The paper presents problems that accompany measuring the thermal conductivity of composite insulating materials with a matrix of polymer resins filled with hollow microspheres used in means of short and long-distance transport. To measure the thermal conductivity with measuring apparatuses available on the market, samples with a specific shape, dimensions and accuracy of workmanship are required. During the drying of water-soluble resins, bubbles and surface deformation develop as a result of water evaporation. Machining the samples to obtain flat and parallel surfaces is not recommended due to the possibility of damage to the microspheres. Moreover, pressing the sample plates with a force that exceeds the permissible pressure for the spheres (3÷5 MPa) in order to reduce air-filled gaps, is not recommended due to the possibility of damaging the coating. Inaccuracy in producing the samples significantly affects the accuracy of the thermal conductivity measurement results by direct methods using the heat flow conducted by the test sample.

Keywords: technical means of transport, thermal conductivity, insulating materials, measurement inaccuracy

PROBLEMY PRZY BADANIU CIEPLNYCH WŁAŚCIWOŚCI IZOLACYJNYCH MATERIAŁÓW KOMPOZYTOWYCH PRZEZNACZONYCH DO TECHNICZNYCH ŚRODKÓW TRANSPORTU

Przedstawiono problemy, które towarzyszą pomiarowi przewodności cieplnej stosowanych w środkach transportu bliższego i dalekiego kompozytowych materiałów izolacyjnych z osnową z żywicy polimerowych wypełnianych pustymi mikrosferami. Do pomiarów przewodności cieplnej dostępnymi na rynku urządzeniami pomiarowymi są wymagane próbki o określonym kształcie, wymiarach i dokładności wykonania. Podczas suszenia żywic wodorozcieńczalnych dochodzi do powstawania pęcherzy i deformacji ich powierzchni w wyniku parowania wody. Obróbka skrawaniem próbek w celu uzyskania płaskich i równoległych powierzchni nie jest zalecana ze względu na możliwość uszkodzenia mikrosfer. Również dociskanie płyt do próbek z siłą powodującą przekroczenia dopuszczalnego nacisku dla sfer (3÷5 MPa), w celu zmniejszenia szczelin wypełnionych powietrzem, nie jest zalecane ze względu na możliwość zniszczenia powłoki. Niedokładność wykonania próbek wpływa istotnie na dokładność wyników pomiarów przewodności cieplnej metodami bezpośrednimi z wykorzystaniem strumienia ciepła przewodzonego przez badaną próbkę.

Słowa kluczowe: techniczne środki transportu, przewodność cieplna, materiał izolacyjny, niedokładność pomiaru

INTRODUCTION

Composite materials have various functions in the construction and operation of machines. At the beginning, composites were used as construction materials due to their increased tensile strength (up to about 30% compared to the matrix material for aluminum alloys). Then they were used as materials in thermal management, including heat dissipation heatsinks of electronic components, e.g. processors in computers. Another application was the lubrication of sliding contacts by embedded solid lubricants. The use of composites as heat and cold insulation materials is newer and less widespread. One of the first applications of cold insulating

composites was the covering of liquid fuel tanks for the space industry [1, 2]. Currently, there are technical means of transport in use for carrying fragile and perishable goods equipped with temperature controlled chambers, including refrigeration. In order to reduce fuel consumption, decrease emissions to the environment and lower transport costs, the latest insulation materials are used to construct means of transport. The simplest form of insulation is foamed polystyrene boards placed between straight plastic covers. Another method is filling a shaped "shell" of the body with polyurethane foam. Using water-soluble composite ma-

materials gives greater possibilities of shaping the body and coating it with insulation. The matrix of such composites are resins, e.g. acrylic, and the phase improving the thermal properties are the microspheres of expanded perlite or other ceramic materials.

At this point, it is tempting to adopt a more general definition of a composite than has been used to date. In most sources the terms "reinforcing" or "strengthening" phase [3-5] are used. In a few works [6, 7], one can find the term "modifying phase", because the addition of, for example, glassy carbon improves the tribological properties, but does not always strengthen the matrix material. The term "filling" phase is also used, which are fillers, e.g. composite adhesives [3]. In composite insulation materials, the basic task of the second phase, called "modifying" by authors, is to reduce the thermal conductivity. If spheres with a higher compressive strength are added to the matrix, they also perform the function of strengthening.

The definition of a composite shows that it must be made of at least two materials with different physical properties, with a clear boundary between the matrix and the strengthening phase. From the thermodynamic point of view, the phase boundaries represent increased resistance to heat transfer. If the composite is composed of a matrix containing nanoparticles, particles or fibers, its thermal conductivity depends on the proportion of the modifying phase and its thermal properties. The larger the number of particles, the larger the number of serial connected barriers for heat flow, which reduces the flow of conducted heat.

One of the basic properties that users of insulating materials want to know is the thermal conductivity defined as heat flow penetrating a barrier of a specified thickness at a temperature difference on both sides of the barrier of 1 K. The standards, many catalogs of insulation material manufacturers as well as publications state thermal conductivity coefficient values determined by λ [W/(mK)] without specifying the measurement temperature. According to the applicable standard [8], the value of λ without specifying the temperature should be understood as the value measured or determined at 20°C. Measuring this property is not simple, and the type of material imposes certain limitations on employing commonly used apparatuses. This article is devoted to problems related to determining the thermal conductivity of composite insulating materials with a matrix of acrylic resins and ceramic spheres as the modifying phase.

MATERIALS AND METHODS

A composite insulating material based on acrylic resin filled with ceramic microspheres made of aluminum oxide was used for the tests. The walls of these spheres have a small thickness, and inside them there is a negative pressure reaching several dozen Pa, which results in a low compressive strength of 3÷5 MPa. The

insulating material is available as a viscous suspension which, after suitable dilution with water, can be applied by brush, roller or spray. For thermal conductivity tests, samples with specific shapes and dimensions are required. A rectangular plate with a thickness of 20 mm and dimensions of 300x200 mm is required for measuring in a plate apparatus. Producing such a plate with a water-soluble material with an acrylic matrix requires using a mold, repeated application and drying of the layers. Making the bottom layer with the required smoothness and flatness is not a problem because the resin is poured onto a flat plate. Problems begin when producing the top layer. Water evaporation (causing the formation of bubbles, Fig. 1) and acrylic cross-linking reactions make the surface uneven, which means that the sample does not adhere to the measurement plates and air-filled gaps are formed. Machining the sample surface is not recommended due to the possibility of destroying the spheres with cutting forces and tool pressures.



Fig. 1. Sample for thermal conductivity measurements with visible surface defects

Rys. 1. Próbkę do pomiaru przewodności cieplnej z widocznymi defektami powierzchni

The view of the sample for thermal conductivity measurement is shown in Figure 1, its surface without machining in Figure 2a and after grinding in Figure 2b. Grinding the surface of the sample causes damage to the spheres in the surface layer of sample and filling of their interior with the grinding products, changes the thermal conductivity because the damaged spheres influence the heat penetration into the material.

The thermal conductivity coefficient (λ) of the composite coating was determined with an HFM 436 Lambda apparatus for testing thermal insulation materials. A diagram of the apparatus is shown in Figure 3. This apparatus allows the thermal conductivity of materials to be measured with a value of λ to 1.0 W/(mK). The apparatus was calibrated in accordance with international standards ASTM, C 518, ISO 8301, ISO 8302, JIS A1412 and DIN EN 12667, DIN EN 12939, DIN EN 13163.

The measurement was carried out in the temperature range from -20 to 80°C (sample temperature) every 10 K. The temperature measurement interval was ad-

justed to the average temperatures prevailing inside vehicle refrigerators and on their surface on hot days. These conditions are similar to the use of insulation on central heating and hot water transport pipes during winter (-20°C outside and 80°C inside the pipe). The temperature difference between the plates was 15 K. A certified glass fiber sample (NIST SRM 1450d) was used for calibration.

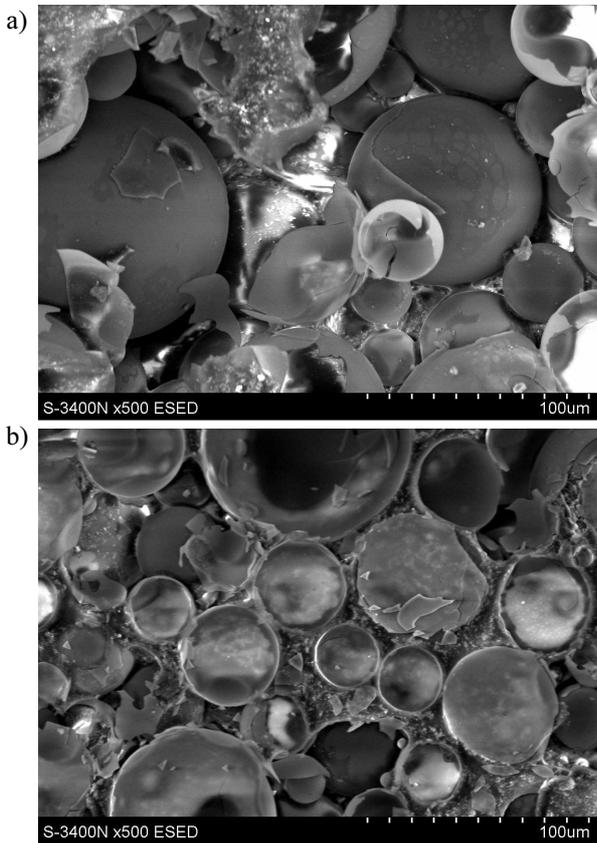


Fig. 2. View of insulating composite coating surface before (a) and after grinding (b) (a - spheres on resin background, b - damaged spheres filled with grinding products)

Rys. 2. Widok z powierzchni izolacyjnej powłoki kompozytowej przed szlifowaniem (a) i po (b) (a - widoczne sfery na tle żywicy, b - uszkodzone sfery wypełnione produktami szlifowania)

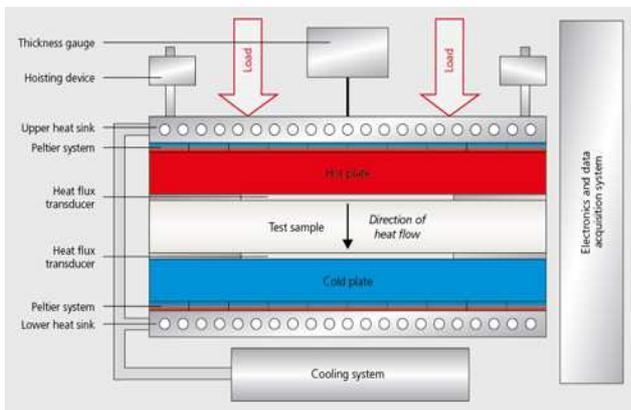


Fig. 3. Diagram of two-plate apparatus for measuring thermal conductivity
Rys. 3. Schemat dwupłytkowego urządzenia do pomiaru przewodności cieplnej

The measurement was carried out twice. After the first measurement, the sample was removed from the apparatus and inserted again. The surfaces of the test sample ($200 \times 300 \times 20 \text{ mm}$) were not perfectly parallel. As a consequence, air gaps formed between the measurement plates and the sample (Fig. 5), which could have influenced the obtained measurement results.

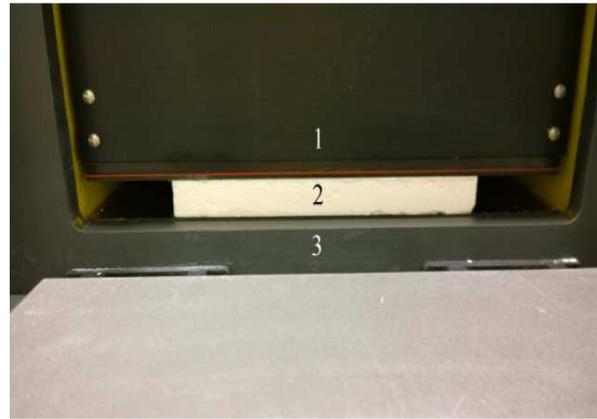


Fig. 4. Composite sample (2) placed between hot plate (1) and cold plate (3)
Rys. 4. Próbkę kompozytową (2) umieszczoną między płytą gorącą (1) a zimną (3)



Fig. 5. Air gap between hot plate and sample influencing measurement accuracy
Rys. 5. Szczelina powietrzna między płytą gorącą a próbką wpływająca na niedokładność pomiaru

RESULTS AND THEIR DISCUSSION

The dependence of the value of the thermal conductivity coefficient (λ) on the temperature in the range from -20 to 80°C is shown in Figure 6. In order to assess the effect of the accuracy of sample preparation for measuring thermal conductivity in the plate apparatus, the measurement results were compared with the thermal conductivity values determined by the comparison method at 70°C using a closed heat source [9, 10].

Comparative measurements were made for coating thicknesses (th) 1.2, 2.4, 3.6 and 4.8 mm. The thermal conductivity of a 20 mm thick coating ($\lambda = 0.0327$) was determined by interpolation with a logarithmic equation

(1) describing the measurement results (coefficient of determination $\alpha = 0.983$; correlation coefficient $R = 0.991$; standard deviation $\sigma = 0.00032$).

$$\lambda = 0.00063983705 + 0.0058545862 \ln(th) \quad (1)$$

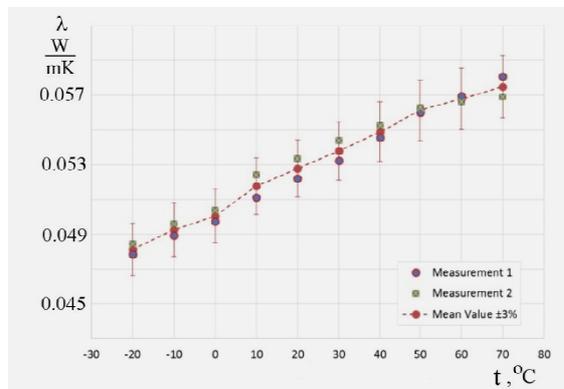


Fig. 6. Dependence of measured thermal conductivity value (λ) of composite coating on temperature (t)

Rys. 6. Zależność zmierzonej wartości przewodności cieplnej (λ) powłoki kompozytowej od temperatury (t)

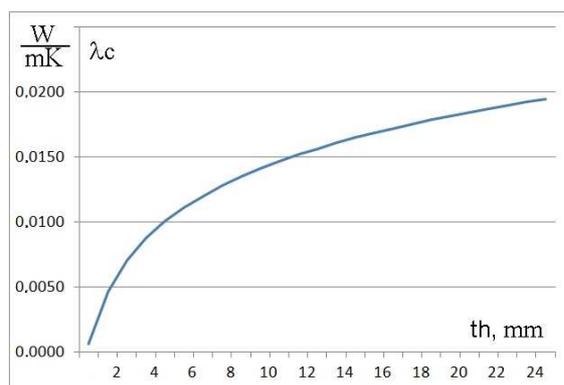


Fig. 7. Dependence of calculated thermal conductivity (λ_c) on thickness (th) of composite coating

Rys. 7. Zależność obliczeniowej przewodności cieplnej (λ_c) od grubości powłoki (th)

Comparison of the diagrams from Figures 6 and 7 shows that the thermal conductivity value measured with the plate apparatus ($\lambda = 0.057$) is 74% higher than that determined by the comparative method ($\lambda = 0.018$). There may be several reasons for this discrepancy. The first, already mentioned, is the inaccuracy in producing the plate samples from the studied material, resulting in gaps between the measuring plates and the tested sample. The second one may be the different structure inside the 20 mm thick sample that hinders the evaporation of water, which results in the binding of water and

resin and partial absorption by the microspheres. The Al_2O_3 spheres after sorption of water are hydrated.

The samples for comparative testing had a maximum thickness of 4.8 mm and were applied in 1.2 mm thick layers and dried after applying each layer, which facilitated the evaporation of water.

CONCLUSIONS

Measuring the thermal conductivity of composite insulation materials with a matrix of water-soluble acrylic resins containing hollow ceramic microspheres creates many problems both in selecting the measurement method and apparatus as well as in preparing the samples. Making rectangular plates with the dimensions 300x200x20 does not always give flat and parallel surfaces, which during measurements results in gaps and increases measurement errors. The thermal conductivity coefficient values of the same material determined by the comparative method using a closed heat source and using a plate apparatus may differ by 60%.

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