



Mechanical and thermal properties of epoxy composite thermal insulators filled with silica aerogel and hollow glass microspheres

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Abstract. Hydrophobic silica aerogel powder and hollow glass microspheres (HGM) were used as fillers for an epoxy adhesive to improve its thermal insulator properties. At 50 wt% of HGM content, the thermal conductivity of the HGM/epoxy composite decreased from 0.182 W/m·K to 0.104 W/m·K. The aerogel/epoxy composite, on the contrary, showed a slight increase in its thermal conductivity, most probably due to the filling of aerogel pores with the epoxy adhesive. Furthermore, it was shown that adhesion values of epoxy composites increase with the addition of aerogel and decrease when HGM was used as the filler material. Realistic numerical finite element method simulations revealed an increase in thermal isolation properties for both HGM/epoxy and aerogel/epoxy composites.

Key words: thermal insulator, epoxy composite, aerogel, hollow glass microspheres, adhesion, polymer testing methods and devices, finite element method.

1. INTRODUCTION

Zero-energy buildings manifest a resourceful combination of construction materials and design in order to effectively manage the household's energy consumption and exchange with the outside environment. For the latter, windows have posed one of the most crucial challenges, and extensive research has been done on improving the insulating properties of glass and glass packets by adding special coatings, using novel materials, and creating layered structures [1–3]. Recent progress in this field has been increasingly profound, as various types of energy efficient glazings such as low-emissivity glazings have been developed. A thorough insulation of window frames whilst keeping their standard thickness combined with good mechanical durability has proven a tough challenge as well. Although decent progress has been made, there is still some room for improvements,

especially in the case of metal frames, where it is essential to isolate the outside and inside parts of the frame with an effective thermal insulator that needs to be mechanically durable at the same time. Here, one possibility is substituting the conventional thermal insulators such as Styrofoam and wool with an adhesive that has a comparable thermal conductivity but superior mechanical durability, thus making the frame technologically less sophisticated. Most feasibly, lower thermal conductivity is achieved by adding micro- or nano-structured materials with excellent thermal insulating properties to conventional adhesives [4,5].

One of the most promising nanostructured isolation filler materials is silica aerogel [4,6–10]. Silica aerogel is a perfect candidate due to its excellent physical properties including a high specific surface area (500–1200 m²/g), a low density (0.003–0.1 g/cm³), a low dielectric constant, a high porosity (>99%), and a low thermal conductivity (0.013–0.04 W/m·K). Therefore, silica aerogel has many potential applications in

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electronics, chemistry, and especially in thermal insulation. However, the use of silica aerogel as an insulation material is limited because due to its brittle and fragile nature it is prone to disintegration already under relatively low external forces. In addition, silica aerogel is an expensive filler material due to the need for sophisticated industrial production equipment and the long drying process with supercritical fluids [9]. Finally, conventional silica aerogel is hydrophilic and tends to absorb moisture, thereby losing its superior heat insulation properties. In fact, loss of heat insulation efficiency due to absorbed moisture is a common problem also for wool and Styrofoam. To counter this problem, we chose to use hydrophobic aerogels in our work. For making a hydrophobic aerogel, the silanols (SiOH) in the conventional aerogel are treated with appropriate chemistry (e.g. alkyl terminated silanes).

Alternatively, hollow glass microspheres (HGM) could be utilized as fillers for thermally insulating epoxy composites [5,11–14]. Similarly to silica aerogel, HGM have a low density, a low dielectric constant, and excellent sound and thermal insulation properties. But contrary to silica aerogels, HGM exhibit a high specific compressive strength and stiffness due to the near perfect spherical shape of the microspheres [15]. Moreover, encapsulation of gas inside closed rigid spheres prevents them from filling with the epoxy adhesive, as is the case for aerogel/epoxy composites [16,17]. In addition, the manufacturing cost of HGM is significantly lower compared to silica aerogels, making them a more cost-efficient material.

Mechanical properties of epoxy composites could be further improved by incorporating a second phase of dispersed rubbery particles into the cross-linked polymers [18]. At the same time, rubbery particles only increase the fracture toughness of epoxy while glass transition temperature and solvent stability decrease. To retain the thermal stability and increase the fracture toughness, epoxy adhesives can be modified with thermoplastics [19]. Balasubramanya and Natarajan showed that adding polymethylmethacrylate (PMMA) to epoxy resin increases its glass transition temperature and tensile strength [20]. To the best of our knowledge, thermal conductivity of such composites has not been measured.

In the present study, the influence of silica aerogel and HGM fillers at different contents on the thermal conductivity and adhesion of epoxy composites are studied and compared. In addition, the influence of PMMA on the properties of epoxy composites is determined. Scanning electron microscopy (SEM) is used to evaluate the microstructure of the composite and the dispersion of the filler material. Finite element modelling (FEM) simulations are used to support the experimental results.

2. MATERIALS AND METHODS

2.1. Preparation of epoxy composites

Epoxy composites were prepared by adding a pre-determined amount of hydrophobic silica aerogel powder (SIL-16-AP, Active Aerogels) or hollow glass microspheres (Q-CEL 300, Potters) to an epoxy adhesive (EPO-TEK 310M-1, Epoxy Technology). The aerogel powder has a density of 0.11 g/cm^3 and a thermal conductivity of $0.025 \text{ W/m}\cdot\text{K}$ as reported by the manufacturer. The HGM have a density of 0.12 g/cm^3 and particle size $5\text{--}200 \text{ }\mu\text{m}$.

First, epoxy resin (part A) was heated to approximately $80 \text{ }^\circ\text{C}$ to decrease its viscosity and then the filler material was gradually added under vigorous stirring using a magnetic stirrer (MSH-20D-Set, Witeg Labortechnik GMBH) to produce a homogeneous mixture. After that, the hardener (part B) was added according to the mix ratio (10 : 5.3) provided by the adhesive manufacturer. Finally, the composite was treated for 10 min in an ultrasonic bath to remove any air bubbles produced during the stirring. Samples of pure epoxy adhesive, 5 wt%, 10 wt%, 20 wt%, and 30 wt% aerogel/epoxy composites and 5 wt%, 10 wt%, 20 wt%, 30 wt%, 40 wt%, and 50 wt% HGM/epoxy composites were prepared (Fig. 1). In addition, PMMA (445746, Sigma-Aldrich) with an average molecular weight of 350 000 was added to improve the mechanical properties of the epoxy composite. Then 5 wt% (with respect to epoxy resin) of PMMA was dissolved in epoxy resin by stirring at $80 \text{ }^\circ\text{C}$ for 12 h or until the PMMA was completely dissolved. After that, the filler and hardener were added using the same steps as described above.

2.2. SEM characterization

Before mixing, aerogel powder and HGM were imaged by a high-resolution SEM (Helios Nanolab 600, FEI).



Fig. 1. Samples for thermal conductivity measurement: HGM/epoxy composites (top row), aerogel/epoxy composites (middle row), HGM/PMMA/epoxy composites (bottom row).

Different SEM (Vega II, Tescan) was used to study the microstructure of the composite material and qualitatively evaluate the dispersion of filler materials. First, a cross-sectional slice was cut from the sample with a razor blade. The slice was then fixed to a SEM stub with a carbon tape. Finally, to avoid charging in SEM, a thin film of 20 nm was deposited on all samples by a sputter coater (SC7640, Quorum Technologies) with a gold-palladium target.

2.3. Measurement of thermal conductivity

The thermal conductivity measurement of epoxy composites is based on the heat flow meter method [21]. The epoxy composite was placed between two copper cylinders and the uniaxial heat flux through the sample was measured. One of the cylinders was immersed into a temperature-controlled water bath and kept at constant temperature of about 50 °C. A magnetic stirrer was used to heat the water bath and measure its temperature. A second cylinder (receiving) with known dimensions and mass was equipped with Pt100 thermoresistor to precisely measure the temperature change of the cylinder. The Pt100 signal was recorded using a data logger (PT-104, Pico Technology). Both the cylinders and the sample were thermally insulated using blue Styrofoam. A good thermal contact between the cylinders and the sample was obtained by applying a thermal compound (MX-2, Artic) at the interface. In addition, a 4 N load was applied on the receiving cylinder to ensure a good mechanical contact. Thermal conductivity measurements were carried out three times for each sample and for every filler concentration; three different samples were measured.

2.4. Measurement of adhesion

Adhesion was measured with a home-made pulling test setup by registering the force along the pulling direction with a 100 kg load cell sensor. The thallus (Fig. 2) for studying the lateral adhesion force consists of two identical s-shaped 2 mm thick and 11.5 mm wide anodized aluminium strips (1) with holes (2) for fixing

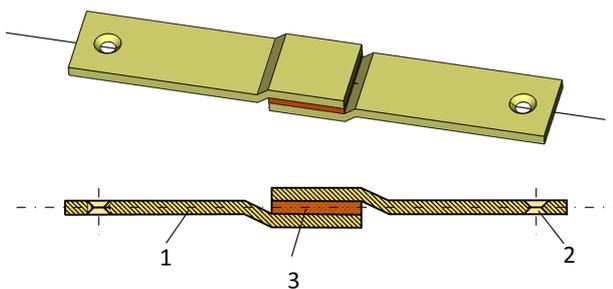


Fig. 2. A sketch of the thallus for studying lateral adhesion of the glue.

the thallus to the pulling device. To apply the force strictly along the central line the holes (2) have phases. The overlapping area of the pieces forms a square (3). The strips were fitted into a rig, the epoxy composite was added between the strips and then placed into an oven for 2 h at 65 °C for hardening. The rig was removed and the plates were fitted into the pulling test setup. The pulling direction was parallel to the plate surfaces. The rupture force was measured.

2.5. FEM simulations

The thermal properties of the composites were modelled using the FEM environment (Comsol Multiphysics). As an input geometry, circular particles with the diameter range measured from SEM images were randomly distributed in a 2D epoxy matrix. The volume content of the fillers was varied in the range of 10–70 vol%, which corresponds to approximately 1–20 wt% of the filler material. The static conditions were simulated and compared to the experimental results. As a result of the simulations, the thermal conductivity dependence on filler concentration was obtained.

3. RESULTS AND DISCUSSION

3.1. SEM characterization

SEM images of aerogel powder and HGM before incorporation into the epoxy adhesive are shown in Fig. 3. Aerogel powder was agglomerated and had bead sizes between 0.1 and 5 µm. SEM images showed a large size distribution of HGM from a few micrometres to 200 µm. Some of the larger spheres were fractured or even broken into small pieces. Debris of the broken HGM could be observed on other spheres. In addition, it was interesting to note that some of the larger spheres were filled with smaller spheres.

All the epoxy composite samples made for thermal conductivity measurements were afterwards imaged by SEM. A selection of the SEM images are shown in Fig. 4. For aerogel/epoxy composites, it can be seen that the aerogel powder is uniformly dispersed into the epoxy matrix and at 5 wt% (Fig. 4b) only a few beads are agglomerated. At 30 wt% (Fig. 4c) aerogel beads are mostly in contact with one another forming larger structures in the epoxy matrix. SEM images reveal that most of the larger HGM are broken inside the epoxy matrix (Fig. 4e,f). At the same time, most of the smaller spheres are still intact. With the increase of HGM wt%, spheres start to form structures with pores and voids inside the epoxy matrix. The HGM are fairly uniformly distributed inside the epoxy matrix. Similar observations were made for HGM/PMMA/epoxy composites (Fig. 4d).

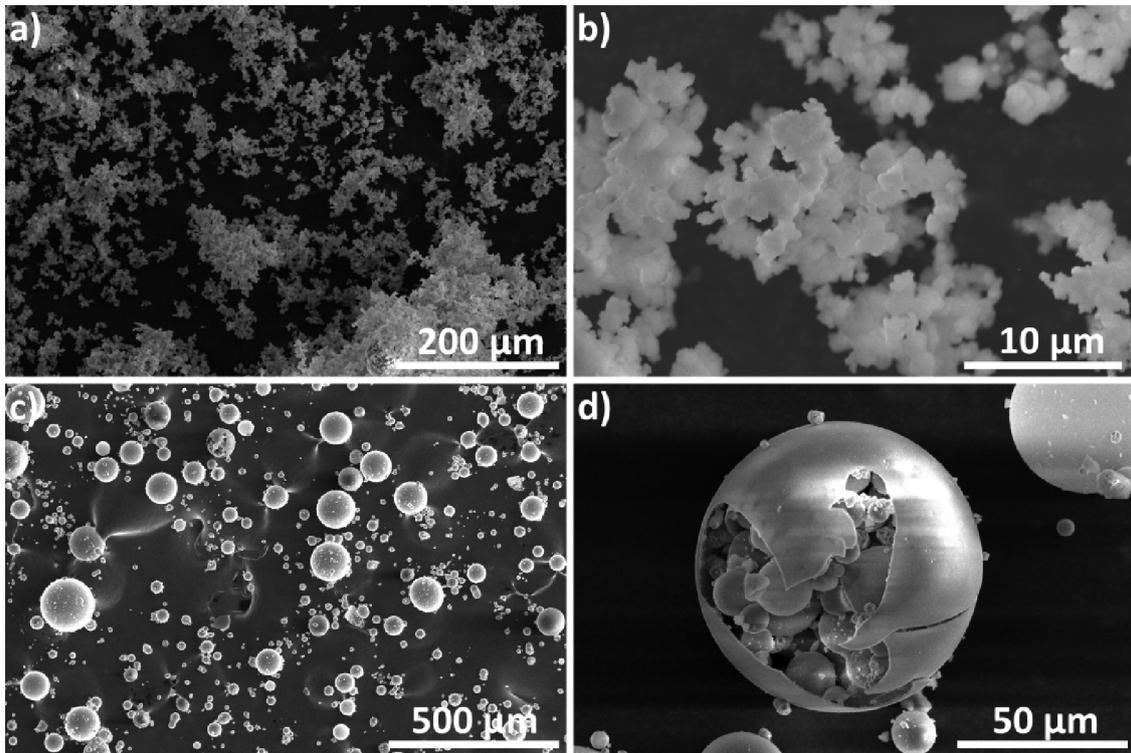


Fig. 3. SEM images of aerogel powder (a, b) and HGM (c, d).

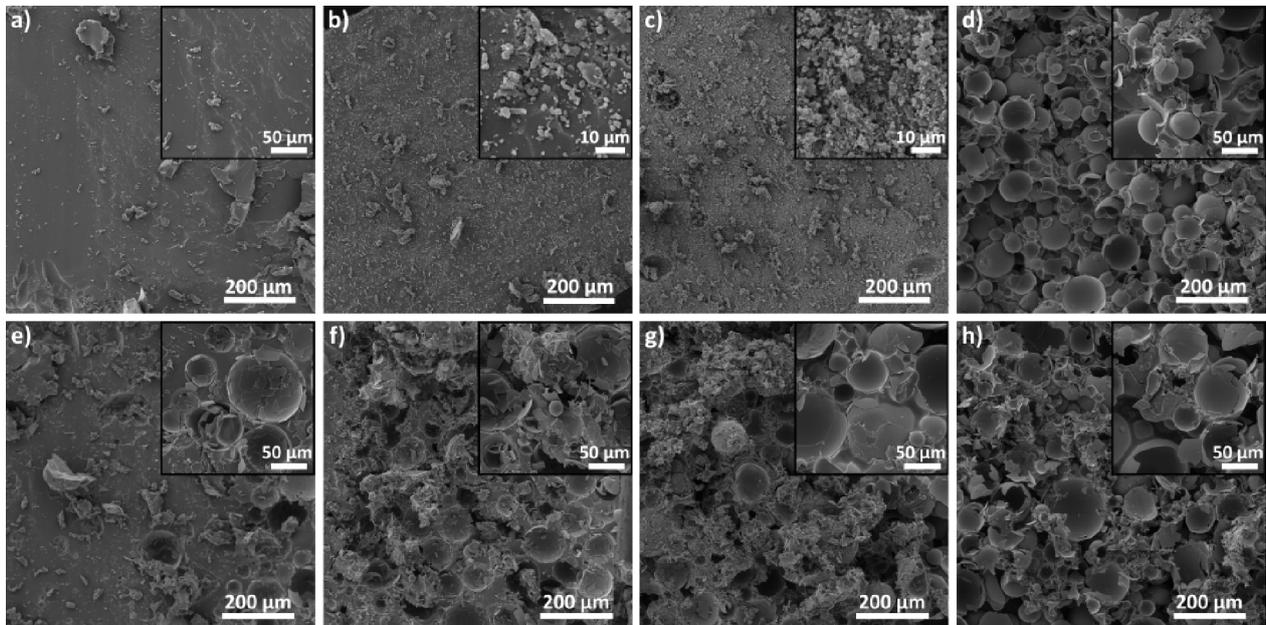


Fig. 4. SEM images of epoxy adhesive (a), 5 wt% (b) and 30 wt% (c) aerogel/epoxy composite; 50 wt% HGM/PMMA/epoxy composite (d); 5 wt% (e), 10 wt% (f), 30 wt% (g), and 50 wt% (h) HGM/epoxy composite.

3.2. Thermal conductivity

For measuring thermal conductivity, the epoxy composite was placed into a mould made by cutting holes of 25 mm in diameter into a 1 mm thick Teflon sheet. The mould was sandwiched between two Teflon sheets and left under a load to cure for 24 h. Thermal conductivity measurements were carried out on all the prepared samples. Figure 5 shows the obtained values for different filler concentrations. The HGM/epoxy and HGM/PMMA/epoxy composite thermal conductivity values show a similar behaviour. With the increase of the filler concentration the thermal conductivity decreased almost exponentially. The epoxy composite with only PMMA showed a slightly higher thermal conductivity compared to the pure epoxy sample. This can be explained by the higher thermal conductivity of the additive. For 50 wt% of HGM, both epoxy composites showed similar values. All in all, addition of HGM decreased the thermal conductivity of the epoxy adhesive almost two times at 50 wt%: from 0.182 W/m·K to 0.104 W/m·K.

The aerogel/epoxy composites exhibited a completely opposite trend. Increase in the aerogel powder concentration led to a slight increase in thermal conductivity. At first, this result seems contradictory as the thermal conductivity of the aerogel powder is an order of magnitude lower than that of the epoxy adhesive. Therefore, the aerogel/epoxy composite should have a lower thermal conductivity value compared to the pure epoxy sample. At the same time, preservation of aerogel

pores is the most significant physical factor in determining the thermal conductivity of the aerogel/epoxy composite [9]. The increase of the thermal conductivity value could mean that some or even most of the aerogel pores are filled with the epoxy adhesive [4,6]. A filling of the aerogel pores was also observed during the mixing process where the volume of the aerogel decreased by 87%. High shrinkage means that it was not possible to produce samples with more than 30 wt% of aerogel powder.

3.3. Adhesion

Adhesion measurements were carried out for pure epoxy and epoxy composites with the filler content between 10 and 50 wt%. Three measurements were performed for each sample. The rupture force was divided by the area covered with glue to calculate the maximum stress before failure.

For the aerogel/epoxy composites the adhesive properties started to improve with increasing the aerogel concentration, reaching more than a two times better performance than pure epoxy (Fig. 6). For the HGM/epoxy composites the adhesive properties decreased with higher microsphere concentrations. Adding PMMA to epoxy increased the adhesion force by 14% compared to pure epoxy. Adding HGM to PMMA/epoxy composites again led to a decrease of adhesion but for lower HGM concentrations, e.g. 20 wt%, the adhesion values were still higher than for pure epoxy.

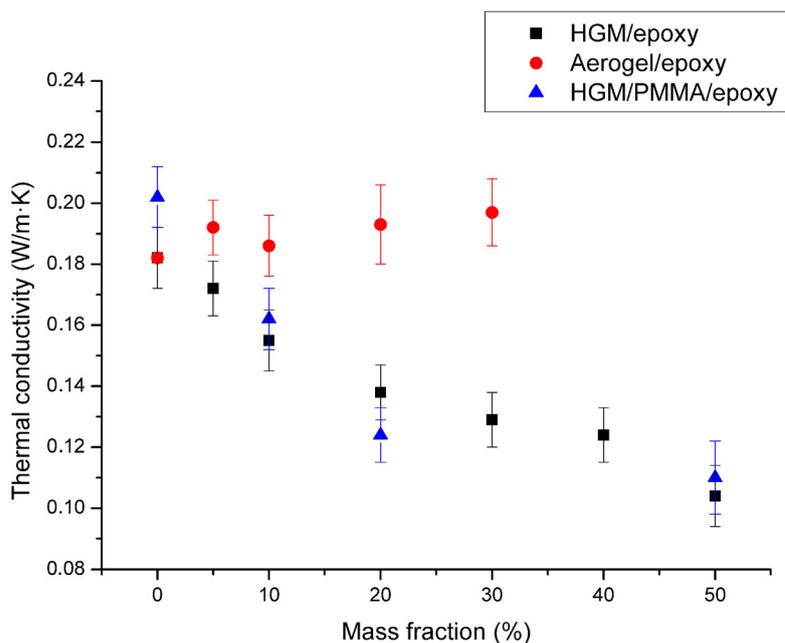


Fig. 5. Thermal conductivity of different epoxy composites dependent on the mass fraction of the filler material.

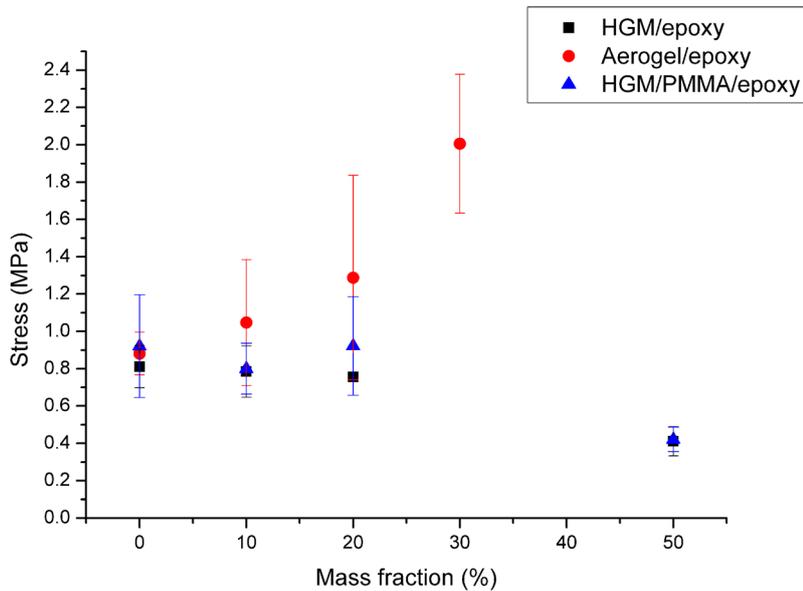


Fig. 6. Maximum stress before breaking of contact for different epoxy composites dependent on the mass fraction of the filler material.

3.4. FEM modelling

Numerical FEM simulations were carried out on both of the composite structures with a filler content of 10–70 vol%. The model consisted of a random distribution of beads within a rectangular epoxy matrix element with a side length of 0.1 mm for the aerogel system and 1 mm for the HGM system. The distributed beads had sizes in the same range as measured from SEM micrographs. As a result, a realistic composite structure of the beads epoxy was obtained.

A number of parameters for correct simulations have to be taken into account. First, as the thermal conductivity of the pure HGM was not provided by the manufacturers, a pre-simulation for calculating this parameter was

conducted. The measured thermal conductivity for the HGM/epoxy composite at 20 wt% was taken as a reference. In the pre-simulation, the thermal conductivity of the HGM was varied to obtain the same composite conductivity as measured in the experiments. The value obtained was approx. 0.11 W/m·K. The thermal conductivity for the aerogel particles was 0.025 W/m·K. At the opposite sides of the rectangle boundary conditions of constant temperature, similar to those achieved in the experiments, were used to obtain a temperature gradient through the material and calculate the thermal conductivity. In the simulations, the filler content was changed with a step of 10 vol%. The results can be seen in Fig. 7a. A clear linear dependence of the thermal conductivity vs concentration can be seen, as was

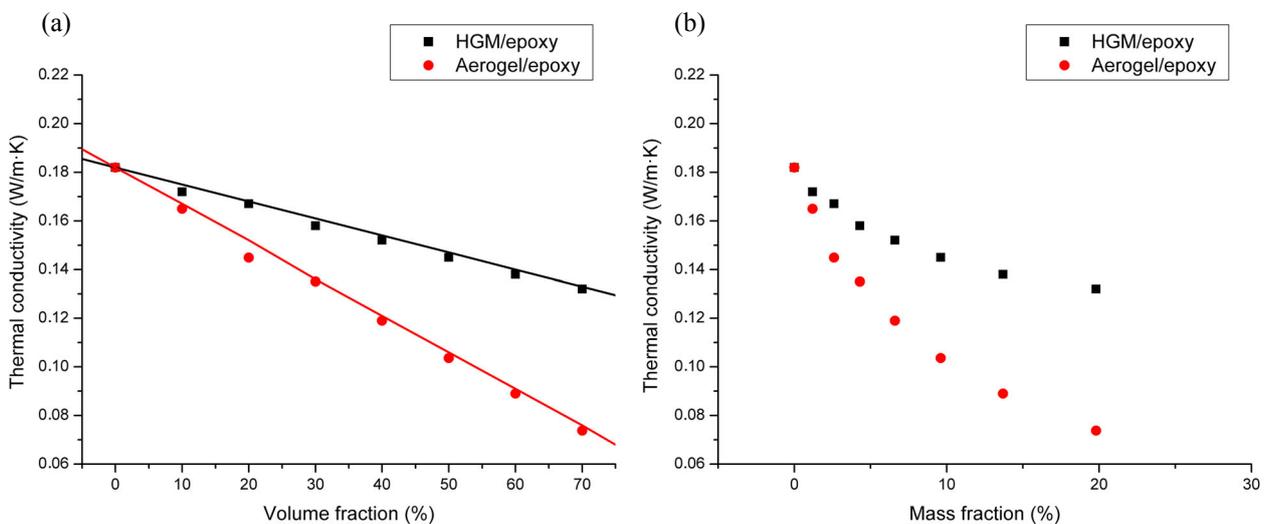


Fig. 7. Results of FEM simulation for thermal conductivity of epoxy composites dependent on volume (a) and mass fraction (b).

expected. Figure 7b shows the same results vs the mass fraction of the filler material.

The simulations of the aerogel/epoxy composite show that in ideal conditions, the composite material has superb thermal insulating properties, indicating that these could be achieved only if the surface structure of the aerogel particles could be modified to an extent that unwanted interactions with the epoxy were diminished. For the HGM/epoxy composite, the experimental and theoretically calculated values are in a good fit. Results indicate that the theoretical model applied is able to realistically predict thermal properties of different composite materials and its applicability to other similar structures is also a possibility. This is true in cases where unwanted chemical and structural interactions do not take place and can be neglected, as in the case of HGM/epoxy composites.

4. CONCLUSION

In this work, silica aerogel and HGM were used as fillers to improve the thermal insulating properties of epoxy adhesives. SEM images showed that both filler materials were uniformly distributed inside the epoxy adhesive. Measurements of thermal properties revealed that an increase of the HGM content in the epoxy composite decreased and of the aerogel content increased the thermal conductivity. The increase of the thermal conductivity of the aerogel/epoxy composite was attributed to the filling of aerogel pores with the epoxy adhesive. In addition, incorporation of PMMA at moderate concentrations did not significantly change the thermal insulating properties of the epoxy composite. Adhesion of the aerogel/epoxy composite increased with the increasing aerogel content while the opposite trend was observed for the HGM/epoxy composites. The FEM simulations were in good agreement with results obtained in HGM/epoxy composite experiments. The developed FEM model could be applied to realistically predict the thermal properties of similar composite materials.

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Räniaerogeeli ja klaasmikrosfääride lisandite mõju epoksiidliimi termilistele omadustele

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On käsitletud hüdrofoobse räniaerogeeli ja õõnsate klaasmikrosfääride (HGM, *hollow glass microspheres*) lisandite mõju epoksiidliimi termilistele omadustele. Komposiidi valmistamiseks lisati liimile erinevas koguses räniaerogeeli või HGM-i, mehaaniliste omaduste parandamiseks omakorda 5% (siin ja edaspidi kõik massiprotsendi järgi) polümetüülmetakrülaati (PMMA) HGM-i sisaldavale komposiidile. Elektronmikroskoopilised analüüsid valmistatud komposiididest viitasid lisandite homogeenisele jaotusele maatriksmaterjalis. Katsekehade soojusjuhtivuslikud omadused määrati soojusvoo mõõtmise meetodika abil. HGM-i lisamine vähendas epoksiidliimi soojusjuhtivustegurit süstemaatiliselt (0,104 W/m·K 50% segu korral, vrd 0,182 W/m·K puhtal epoksiidliimil), samas kui räniaerogeeli täiteainena kasutamine mõnevõrra hoopis suurendas seda (0,197 W/m·K 30% segu korral). PMMA lisamine ei põhjustanud komposiidi soojusjuhtivusteguri märkimisväärset muutumist. Soojusjuhtivusteguri suurenemine viitab epoksiidliimi tungimisele aerogeeli pooridesse. Lisaks määrati mõlema komposiidi liimliite tugevus. HGM-lisand vähendas epoksiidi liimliite tugevust, kuid lisandi mõju püsis minimaalne kuni sisalduseni 20%. Räniaerogeelilisand suurendas epoksiidi liimliite tugevust, kusjuures jällegi muutus trend märkimisväärseks alates 20% sisaldusest, suurenedes 30% lisandi sisalduse korral juba üle poole esialgsest väärtusest (ehk 2 MPa, vrd 0,8 MPa). Ka see tulemus viitab epoksiidliimi tungimisele aerogeeli pooridesse. Lisaks eksperimentaalsetele analüüsidele viidi läbi teoreetilised simulatsioonid (kasutades lõplike elementide meetodikat), määramaks lisandite mõju maatriksmaterjali soojuslikele omadustele. HGM-lisandiga komposiidi puhul andis teoreetiline simulatsioon eksperimentaalse tulemusega üllatavalt hea kooskõla. Räniaerogeelilisandi puhul pakkus teoreetiline simulatsioon sõltuvalt aerogeeli kontsentratsioonist soojusjuhtivusteguri veelgi järsemat langust, mis näitab, et pooride täitumist ära hoides on võimalik luua väga hea isolatsiooniomadustega komposiitmaterjale.

Kokkuvõttes võib öelda, et HGM-lisand võimaldab epoksiidliimi soojusjuhtivustegurit efektiivselt vähendada, andes märkimisväärse efekti juba 20% sisalduse korral, kus selle mõju liimi mehaanilistele omadustele on tühine. Hüdrofoobne räniaerogeel töötlemata kujul ei sobi epoksiidliimi soojusjuhtivusteguri alandamiseks, kuna epoksiidliim imendub aerogeeli pooridesse.