



# Loads transfer across static electrical phase interfaces in silica aerogel/polymethyl methacrylate composites



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## ABSTRACT

Silica aerogel (SA)/polymethyl methacrylate (PMMA) composites with static electrical phase interfaces were prepared to investigate loads transfer across the interfaces. Preparations of SA/polymer hybrid composites were based on the interaction between the positive charges in SA and negative charges in polymer. Characterizations of the SA, polymers, and composites were performed by scanning electron microscopy (SEM), Raman spectroscopy, thermogravimetric analysis (TGA), and so on. A Raman microscope system was employed for the investigation of loads transfer across phase interfaces. The results indicated that the SA/PMMA composites with static electrical phase interfaces and nanoporous structure were obtained. Based on the Raman mapping results, the composite with covalent bonding phase interfaces got the most efficient load transfer ability. The load transfer through the static electrical phase interfaces in composites is also effective. The static electrical phase interfaces that are physical interactions between the silica phase and polymer phase are with high interfacial thermal resistances, and the heat flux could be held in silica phase. It could be achieved with minimal changes to the overall thermal conductivity of the material as possible.

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## 1. Introduction

Silica aerogels (SA) [1] are nanostructured materials, and with the combination of low density, transparency, high surface area, high porosity, and low thermal conductivity [2]. This has made aerogels attractive for thermal and acoustic insulators, catalyst supports and low-k dielectric materials [3–5]. However, their intrinsically low density and fragility greatly complicate handling or processing without catastrophic fracture of the material [6].

Different methods have been reported for the improving of mechanical properties of SA such as structural reinforcement using flexible silica precursors in silica gel backbone, conformal coating of silica back bone via surface cross-linking with a polymer, and dispersing organic or inorganic fibers in the initial sol of SA [7–10]. In most of these works, while the strength of aerogels was improved, and undesirable increases in the solid state density and reduction of the effective surface areas of the material were also observed. Hence, improving the mechanical properties of aerogels is an important fundamental challenge towards achieving the full

potential of these materials for various applications [6]. Ideally, for thermal insulation applications, the reinforcement of SA should be achieved with minimal changes to the overall density and porosity of the material as possible [11,12].

Boday et al. [6] reported that the optimal mechanical reinforcement of aerogels could be achieved by the attachment of higher molecular weight organic macromolecules with low grafting densities onto SiO<sub>2</sub> surfaces without significantly increasing the density of the material. Compounding the silica network with polymer can be achieved by different type of interfacial interaction of secondary silica particles with appropriate functionality on the organic polymers [13]. Depending on the chemical relationship between the polymer and surrounding silica network, polymer/SA composites are placed into two categories: Class I hybrid composite aerogels (the composites that are formed as a result of physical interactions, like Van der Waals forces, electrostatic forces, or hydrogen bonding, between the organic and inorganic phases) and Class II hybrid composite aerogels (the composite in which the interfacial bonding between the organic phase and silica is based on covalent chemical bonds) [14,15].

Although, it has been proved that compounding of silica backbone with chemically bonded polymer is an effective way for increasing mechanical strength, whether the thermal conductivity

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of aerogels would increase is still unknown. Losego and Nitzan's research [16–18] show that the strength of covalent chemical bonds directly controls phonon heat transport across an interface. The transitioning from Van der Waals to covalent bonding increases interfacial conductivity by ~80%. If we incorporate macromolecules onto the surface of SA networks via a covalent bond to obtain polymer reinforced aerogel (a Class II composite), the strong chemical bonds could be a “highway” of heat flux [19]. This would lead to the thermal transmission from SA skeleton to polymer phase based upon the Minimum Thermal Resistance law. How about the Class I composites? Physical interactions between the silica phase and polymer phase are with high interfacial thermal resistances, and the heat flux could be held in silica phase. It could be achieved with minimal changes to the overall thermal conductivity of the material as possible [20,21].

A number of techniques have been developed to assess the strength of the interface between inorganic phase and polymer matrix materials [22–25]. Raman spectroscopy has been used widely to follow stress transfer between the matrix and reinforcement in a wide range of polymer-based composites [26]. In general, the shift in the Raman band rate is proportional to the stress or strain in the reinforcement due to changes in bond length. As the bond elongates, which on the macroscale is equivalent to tensile strain, the Raman wavenumber generally undergoes a red-shift that reverses on unloading [27].

In this research, we prepared SA/polymer composites based on the interaction between the positive charges of SA and negative charges in the polymer [28–31]. The construction of phase interfaces and the mechanisms of phase interfaces on the reinforcement and thermal conduction of the composites were investigated.

## 2. Experimental

### 2.1. Materials

Methacryloxyethyltrimethyl ammonium chloride (MTC) was supplied by Sigma-Aldrich Co. LLC. MMA was purchased from Sigma-Aldrich Co. LLC. and purified by the standard treatment with 5% aqueous NaOH and then deionized water, followed by distillation at normal pressure and finally stored at low temperature prior to use. Benzoyl peroxide (BPO, analytical grade, Beijing Chemical Factory, Beijing, China) was used as initiator. Other reagents were all of analytical reagents.

### 2.2. Preparation of SA

TMOS (0.548 g, 0.00360 mol) in anhydrous methanol (1.8 mL) was mixed with 1 N ammonium hydroxide (0.259 mL) in anhydrous methanol (1.8 mL) to afford a clear colorless solution (3.6 mL) that gelled in 10 min. Silica alcogels were aged at room temperature for 48 h at room temperature followed by aging at 50 °C for 48 h. Subsequent SCD resulted in SA.

### 2.3. Preparation of composites

Mixtures of desired wt% of SA, MTC,  $\gamma$ -methacryloxypropyltrimethoxysilane, with methylmethacrylate monomer (MMA) were taken separately in a three necked round bottom flask and kept in stirring at 60 °C for 30min. This was followed by addition of 1.0 wt% benzoyl peroxide under nitrogen atmosphere. Sonication was continued under nitrogen atmosphere until the polymerization was completed and the suspension turned to solid (4–5 h). The solid was then magnetically stirred using chloroform (CHCl<sub>3</sub>) as a solvent to get a uniform dispersion. The dispersion was cast on a glass plate to get sheets of uniform thickness (~4 mm). The samples

were then stripped of the glass slides. The compositions of the samples are shown in Table 1, and the polymerization reaction is shown in Scheme 1.

### 2.4. Characterization

Examination of the morphology of samples was performed on a Philips XL-30 scanning electron microscope (SEM). The surfaces of samples were sputter coated with a thin layer of gold prior to examination. Zeta potential values of SA, P(MMA-co-MTC) and SA/P(MMA-co-MTC) composites were measured by a Brookhaven Zetasizer (Brookhaven Instruments Ltd., US). Zeta limits ranged from –150 to 150 V. Strobng parameters were set as follows: Strobe delay –1.00, on time 200.00 ms, and off time 1.00 ms. The natural (unbuffered) pH of the aqueous samples was used. Each experiment was repeated at least four times and the mean value was reported. Fourier transformed infrared spectroscopy (FTIR) spectra of the samples were recorded by a Nicolet Magna Nicolet-5DX FTIR spectrometer in the scan range of 4000–400 cm<sup>-1</sup> with a resolution of 4 cm<sup>-1</sup> using KBr disc technique. <sup>1</sup>H nuclear magnetic resonance (<sup>1</sup>H NMR) spectra of PMMA, P(MMA-co-MTC) and SA/P(MMA-co-MTC) were recorded using a UNITY plus-500 NMR spectrometer (Varian, USA) with CDCl<sub>3</sub> as the solvent. Chemical shifts were reported in ppm units with tetramethylsilane as an internal standard. Before FTIR and NMR measurements, SA/P(MMA-co-MTC) composite sample was Soxhlet extracted with refluxing acetone for 48 h, then dried at 60 °C to remove the acetone. Thermogravimetric analysis (TGA) was carried out using a Rigaku TA-50 instrument (Japan). Samples were heated to 500 °C at heating rates of 20 °C/min under air. Mechanical properties were measured using a universal material testing system (M350-20KN, Testometric, UK). Measurements were made at room temperature at a constant crosshead speed of 5 mm/min. Data were taken as averages of at least twenty specimens per sample. The Raman spectra were measured using a Renishaw in Via system confocal Raman microscope system with a Nd: YAG laser source operated at a wavelength of 532 nm. The probing depth was approximately 1–2  $\mu$ m and the diameter of the laser spot was 0.76  $\mu$ m. All Raman spectra were measured at room temperature with fixed exposure time and integration number. For the Raman mapping analysis, the measured spectra were fitted with a Lorentz function, and the peak position and full width at half maximum (FWHM) values were used to create the mapping images. For thermal conductivity, a FP2C Neotim based on the Hotwire method was employed. In this test, specific probe is sandwiched between two plates of the material to be measured, so that the thermal response is an average between the properties of both plates.

## 3. Results and discussion

Evaluation of size distribution and surface morphology of the modified silica aerogel nanoparticles were performed by SEM. Fig. 1 exhibits SEM images of SA and SA/P(MMA-co-MTC). According to

**Table 1**  
Composition.

No.	Sample	SA(wt%)	Reaction ratio		
			MMA	MTC	KH570
A	SA	100	0	0	0
B	PMMA	0	100	0	0
C	P(MMA-co-MTC)	0	100	7	0
D	SA/PMMA	10	100	0	0
E	SA/P(KH570-MMA)	10	100	0	7
F	SA/P(MMA-co-MTC)	10	100	7	0

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