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# Carbon Nanotube Thermal Pastes for Improving Thermal Contacts

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The use of 0.6 vol.% single-walled carbon nanotubes in a poly(ethylene glycol)-based dispersion gave a thermal paste that was as effective as solder for improving thermal contacts. A thermal contact conductance of $20 \times 10^4$ W m$^{-2}$ K$^{-1}$ was attained. An excessive amount of nanotubes (e.g. 1.8 vol.%) degraded the performance, because of conformability loss. The nanotubes were more effective than hexagonal boron nitride particles but were less effective than carbon black, which gave a thermal contact conductance of $30 \times 10^4$ W m$^{-2}$ K$^{-1}$.

**Key words:** Carbon nanotube, polymer, paste, dispersion, thermal contact, thermal conductance

## INTRODUCTION

Carbon nanotubes[1] have been investigated for applications including flat panel (field emission) displays,[2–4] nanoelectronic devices,[5–7] gas sensing,[8] structural composites,[9,10] tribology,[11] damping,[12] actuation,[13] and microelectromechanical devices.[14] Although the thermal conductivity of carbon nanotubes is high,[15–17] that of carbon nanotube composites is low,[18,19] because of the interface between the nanotube and the matrix.[20–23] The most promising thermal application of carbon nanotubes is their use in thermal interface materials, i.e. materials for improving thermal contacts, for example that between a microprocessor and the heat sink of a computer.[24–30] Thermal management[31] is critically needed in microelectronics for the purpose of dissipation of the heat which limits the performance, reliability, power, and further miniaturization of electronics.

Materials for thermal management fall into two categories—materials for thermal conduction (e.g. materials for heat sinks) and thermal interface materials (e.g. thermal pastes).[32–35] Much more research has been conducted on the former than the latter. However, the latter is critically needed in practice, because a poor thermal contact between a heat source and a heat sink would make the use of an expensive heat sink not worthwhile.

The paper is focused on the development of thermal pastes using carbon nanotubes. Thermal pastes[24,25] are to be distinguished from thermal interface materials in the form of nanotube array coatings.[26–30] A paste is more attractive than a nanotube array coating in its ease of application (for example by screen printing) and feasibility of application to a large variety of surfaces. In contrast, a nanotube array coating is applied by chemical vapor disposition.[26,28–30]

The carbon nanotube thermal pastes of this work differ from those of previous work[24,25] in both the formulation and the performance. Previous work used a liquid-crystal matrix to align the nanotubes.[24] Other previous work used 2 vol.% nanotubes with 30 vol.% nickel particles in polydimethylsiloxane oil.[25] In contrast, the formulation in this work was developed by consideration of enhancing the conformability of the paste. The importance of conformability is explained below. For the purpose of assessing the performance, this work also provides a comparative evaluation that involves solder and pastes that contain carbon black or boron nitride particles.

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Carbon nanotubes are attractive for thermal pastes because of their high thermal conductivity and small size. The need for a small size stems from the importance of conformability and spreadability in governing the effectiveness of a thermal paste. Because a thermal paste is applied as an extremely thin layer (just enough material to fill the valleys in the surface topography) between the mating surfaces, the number of junctions between adjacent nanotubes needed to form a conductive path is relatively small.

As the mating surfaces that sandwich a thermal paste are not perfectly flat, the air pockets resulting from the asperities must be displaced by the thermal paste. This displacement requires the paste to be highly conformable to the topography of the mating surfaces. In addition, the paste must be highly spreadable, so that it is very thin, ideally with just enough material to displace the air pockets. The thicker is a thermal interface material, the higher is the thermal resistance, and the less effective is the material. Conformability and spreadability can be even more important than thermal conductivity in governing the performance of a thermal paste, when the thermal paste applied is sufficiently thin.[36] The thicker is the paste at the thermal interface, the more important the thermal conductivity within the paste. This work is limited to a small paste thickness of 25 $\mu$m or less and a mating surface roughness of only 0.05 $\mu$m.

That the thermal conductivity within a thermal paste can be less important than the conformability for a small paste thickness has been shown by the exceptionally high performance of carbon black thermal paste.[37] Carbon black is not as thermally conductive as silver, but carbon black thermal paste is more effective than silver thermal paste for a small paste thickness.[38–40] That the thermal conductivity within a thermal paste can be less important than the conformability has also been shown by the fact that the thermal contact conductance is maximum at an intermediate content of the conductive filler (boron nitride[36] or carbon black[37]) in the thermal paste; the conductance decreases as the conductive filler content is increased beyond the optimum, because of a decrease in the conformability (despite the increase in the thermal conductivity within the paste). In this work a similar optimum was found at intermediate carbon nanotube content. Although the thermal conductivity (in W $m^{-1}$ $K^{-1}$) within a material is a well-known attribute concerning the thermal behavior, it is not a good indicator of thermal paste performance. Rather, the thermal contact conductance (in W $m^{-2}$ $K^{-1}$, which is the same as W $m^{-2}$ per degree Celsius) across surfaces that sandwich a thermal paste is a good indicator.

A thermal paste is a dispersion which comprises a vehicle (a liquid) and a thermally conductive material (typically particles). For the paste to be highly conformable and spreadable the vehicle is preferably one of low viscosity and the solid component cannot be excessive.

Carbon nanotube pastes have also previously been investigated for applications other than thermal management. An application relates to the fabrication of field emission displays by screen printing and related thick film processing methods.[41–44] They have also been investigated for optical limiting applications.[45] In addition, the dispersion of carbon nanotubes in polymers has previously been investigated for the purpose of making polymer–matrix composites.[46–54]

A widely used vehicle for thermal pastes is polydimethylsiloxane,[25,55–58] which suffers from a high viscosity. Poly(ethylene glycol) (PEG) of low molecular weight (400 amu)[36] and sodium silicate[59] are more effective than polydimethylsiloxane, because of their relatively low viscosity. This work uses PEG along with dissolved ethyl cellulose,[60] which serves to enhance the dispersion and suspension.[61] Ethyl cellulose has the further advantage of its slight conductivity.[62,63] Compared with the sodium silicate-based pastes, the pastes developed in this work are advantageous in their long-term compliance (mechanical softness), in contrast with the long-term rigidity (mechanical stiffness) of sodium silicate. Compliance is attractive for reducing thermal stress, which can cause thermal fatigue.

The most widely used thermal interface material in microelectronics is solder, which is applied in the molten state. Compared with solder, thermal pastes have the advantage that they do not require heating.

## EXPERIMENTAL METHODS

### Materials

The poly(ethylene glycol) (PEG, or $HO(CH_2CH_2O)_nH$) used as an organic vehicle was PEG 400 from EM Science (Gibbstown, NJ, USA). It had a molecular weight of 400 amu. It was a liquid room temperature and optionally contained ethyl cellulose (Sigma, St Louis, MO, USA) at 2.5 vol.%. The ethyl cellulose was a white powder that was dissolved in the vehicle. Poly(ethylene glycol) dimethyl ether ($CH_3(OCH_2CH_2)_nOCH_3$) (Sigma-Aldrich, Milwaukee, WI, USA), which differed from PEG in the absence of an OH end group, was used for improving the fluidity of the PEG-based paste; it was a liquid at room temperature, with an average molecular weight 250 amu.

The carbon nanotubes used were AP-Grade nanotubes (single-walled carbon nanotubes of diameter approx. 1.4 nm and purity 50–70 vol.%, in the form of 20 nm diameter "ropes" of parallel individual nanotubes) from CarboLex (Lexington, KY, USA). The surfactant Brij 30 (polyoxyethylene(4) lauryl ether, $C_{12}H_{25}(OCH_2CH_2)_nOH$, $n \approx 4$) (Sigma–Aldrich) used was a liquid at room temperature. It served to help the dispersion of the carbon nanotubes.

### Sample Preparation

The surfactant was dissolved in acetone. The carbon nanotubes were then added and mixed in a sealed bottle under magnetic stirring for 12 h to obtain a nanotube dispersion. The surfactant used was 0.5% by weight of the carbon nanotubes.

Ethyl cellulose was dissolved in PEG, with heating and stirring, which was conducted until the liquid became clear. Then poly(ethylene glycol) dimethyl ether was added to the solution and mixed by stirring. This constituted the base of the paste for use with the carbon nanotubes. The base contained 2.4 vol.% ethyl cellulose, 2.9 vol.% poly(ethylene glycol) dimethyl ether, and 94.7 vol.% PEG.

The nanotube dispersion was added to the base of the paste and then mixed in a ball mill for 2 h. The dispersion was then heated in a vacuum oven to remove the acetone and mixed in a ball mill for another 2 h; this resulted in a carbon nanotube-filled paste. Pastes containing different amounts of carbon nanotubes (0.3, 0.6, 1.8, and 3.0 vol.%) were prepared.

### Thermal Contact Conductance Measurement

Different thermal pastes were sandwiched between the flat surfaces of two copper disks (both surfaces of each disk having been mechanically polished by use of 0.05-$\mu$m alumina particles), of diameter 12.6 mm and thickness 1.16 mm for one disk and thickness 1.10 mm for the other disk. The thermal contact conductance between two copper disks with and without a thermal interface material was measured using the transient laser flash method.[36,59,64,65] The pressure on the sandwich was controlled at 0.46, 0.69, or 0.92 MPa, because pressure affects the thermal contact conductance, even for a material which is not resilient.[59] The thickness of the thermal interface material was 25 $\mu$m or less. The uniform distribution of the paste in the plane of the sandwich was made possible by the fluidity of the paste and the use of pressure. The interface material thickness was obtained by subtracting the thicknesses of the two copper disks from the thickness of the sandwich, such that all thicknesses were measured using a micrometer. The interface material thickness for all cases was the same before and after the conductance measurement.

At least three specimens were obtained from each paste and tested by measurement of the thermal contact conductance across copper mating surfaces to determine the reproducibility and scatter of the data. The reproducibility of the data obtained from different specimens from the same paste indicates adequate uniformity of the nanotube distribution in the paste.

For the sake of comparison, solder (applied in the molten state) was also used as a thermal interface material. The solder was tin–lead–antimony (63Sn-36.65Pb-0.35Sb), with activated Rosin flux core. Molten solder at a temperature of 187°C, measured by use of a Type-T thermocouple, was sandwiched between copper disks that had been preheated to the same temperature. This temperature was above the liquidus temperature of 183°C. The heat was provided by a hot plate. The copper–solder–copper sandwich was allowed to cool on the hot plate with the power off under slight pressure. The thickness of the solder was 25 $\mu$m or less.

The finite element software ABAQUS was used to calculate the thermal contact conductance from experimentally obtained temperate–time curves. The calculation[36] assumed no thickness and no heat capacity for the interface between the two copper disks. It also assumed no heat transfer between specimen and environment except for the absorption of laser energy by the specimen. It also assumed the laser energy was uniformly absorbed on the surface of the specimen, that the heat flow was one-dimensional, and that the thermal contact conductance between the two copper disks was uniform. The validity of these assumptions is supported by the calibration result and error analysis given below.

A Coherent General Everpulse Model 11 Nd glass laser with a pulse duration of 0.4 ms, a wavelength of 1.06 $\mu$m and a pulse energy up to 15 J was used for impulse heating. The laser power was adjusted to produce an increase in the temperature of the specimen between 0.5 and 1.0°C. The upper surface of disk #1, which the laser beam hit directly, had been coated with carbon to increase the extent of laser energy absorption relative to the extent of reflection (Fig. 1). An E-type thermocouple (#1) was attached to the back surface of disk #2 to monitor the temperature rise. Another thermocouple (#2) of the same type was put ~30 cm above the specimen holder to detect the initial time when the laser beam was turned on. A National Instruments DAQPad-

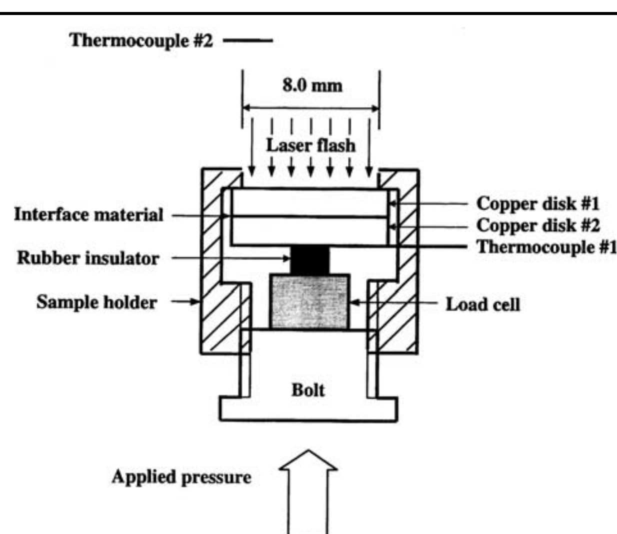


Fig. 1. Experimental set-up for thermal contact conductance measurement.

MIO-16XE-50 data acquisition board with a data acquisition rate up to 20,000 data points per second at 16 bits resolution, with NI-DAQ interface software coded in Visual Basic, was used to monitor the response of both thermocouples simultaneously. A plexiglass holder (Fig. 1) was used to facilitate pressure application. A Sensotec (Colombus, OH, USA) Model 13 load cell was used for pressure measurement. Calibration using a standard graphite specimen was performed before testing each specimen, to ensure measurement accuracy. The data acquisition rate used for each test was adjusted so there were at least 100 temperature data points during the temperature rise.

The experimental error in transient thermal contact conductance measurement consists of random error, because of experimental data scatter, and systematic error, mainly because of the lag of the thermocouple response and partly because of the method used to calculate the conductance from the temperature data. The higher the thermal contact conductance, the shorter the time for the thermocouple temperature rise and the greater is the error. The thermal diffusivity of a standard NBS 8426 graphite disk (thickness 2.62 mm), which had a similar transient temperature rise time as the copper sandwich with the highest thermal contact conductance, was measured before testing each specimen to determine the systematic error, if any. The random error shown by the ± value was determined by measurement of three specimens.

## RESULTS AND DISCUSSION

Table I gives the thermal contact conductance for different thermal interface materials. Included in the comparison are results obtained with the same method for polydimethylsiloxane-based paste, lithium doped PEG-based pastes, sodium-silicate-based pastes, and solder.[36,59]

As shown in Table I, the thermal contact conductance obtained by use of PEG was essentially not affected by addition of ethyl cellulose (2.5 vol.%). Further addition of poly(ethylene glycol) dimethyl ether (2.9 vol.%) reduced the conductance, as also shown in Table I.

The dependence of thermal contact conductance on nanotube content is shown in Table I and Fig. 2. The conductance was increased by the addition of nanotubes such that it reached a maximum value of $20 \times 10^4$ W m$^{-2}$ K$^{-1}$ at an intermediate nanotube content of 0.6 vol.%. Higher nanotube contents (1.8 vol.% and above) resulted in decrease of the conductance. That an intermediate content of conductive solid gave the highest conductance is consistent with previous reports of optimum solid content. For carbon black the optimum is at 1.25 vol.% for a paste based on PEG with 3 vol.% ethyl cellulose[37] and at 2.4 vol.% for a paste based on polyol esters.[66] For hexagonal boron nitride particle (5–11 $\mu$m) pastes based on lithium-doped PEG the optimum is 18 vol.%.[36] The optimum of 0.6 vol.% found in this work for the carbon nanotube is much

**Table I. Thermal contact conductance ($10^4$ W m$^{-2}$ K$^{-1}$) for different thermal interface materials between copper disks at different contact pressures**

| Thermal Interface Material | Contact Pressure (MPa) 0.46 | 0.69 | 0.92 | Ref. |
|---|---|---|---|---|
| PEG | 11.0 ± 0.3 | – | – | 36 |
| PEG + ethyl cellulose (2.5 vol.%) | 10.8 ± 0.1 | 11.1 ± 0.2 | 14.3 ± 0.9 | This work |
| PEG + ethyl cellulose (2.5 vol.%) + poly(ethylene glycol) dimethyl ether (2.9 vol.%)* | 9.4 ± 0.6 | 9.7 ± 0.6 | 9.7 ± 0.4 | This work |
| Paste with 0.3 vol.% nanotubes | 13.5 ± 0.2 | 13.8 ± 0.3 | 14.1 ± 0.4 | This work |
| Paste with 0.6 vol.% nanotubes | 19.9 ± 0.7 | 23.6 ± 0.6 | 20.4 ± 0.6 | This work |
| Paste with 1.8 vol.% nanotubes | 11.3 ± 0.5 | 12.2 ± 0.4 | 12.5 ± 0.4 | This work |
| Paste with 3.0 vol.% nanotubes | 3.9 ± 0.2 | 4.3 ± 0.2 | 5.2 ± 0.3 | This work |
| PEG + ethyl cellulose (3.0 vol.%) + carbon black (1.25 vol.%) | 29.9 ± 0.8 | 28.4 ± 2.1 | 29.6 ± 1.9 | 66 |
| Butyl ether + ethyl cellulose (40 vol.%) + carbon black (0.20 vol.%) | 27.4 ± 2.8 | 28.4 ± 2.8 | 28.0 ± 1.6 | 66 |
| Sodium silicate + BN (16.0 vol.%) | 18.2 ± 0.7 | 17.8 ± 0.7 | 17.7 ± 0.5 | 59 |
| Silicone + BN (16.0 vol.%) | 10.9 ± 0.2 | 12.0 ± 0.2 | 13.4 ± 0.1 | 59 |
| Silicone | 3.08 ± 0.03 | 3.34 ± 0.05 | 4.05 ± 0.05 | 59 |
| PEG + BN (18 vol.%) + Li salt (1.5 wt.%) + water + DMF | 18.9 ± 0.8 | – | – | 36 |
| Silicone + ZnO | 12.2 ± 0.6 | 13.7 ± 0.7 | 13.5 ± 0.2 | 59 |
| None | 0.68 ± 0.01 | 0.88 ± 0.04 | 0.99 ± 0.03 | 59 |
| Solder | 20.1 ± 0.6† | – | – | This work |

All data in this table were obtained using the same testing method.
*Paste base used for all nanotube pastes.
†No contact pressure was applied during testing.

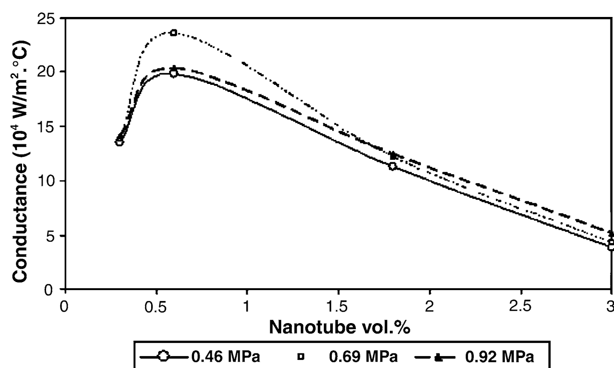


Fig. 2. Variation of the thermal contact conductance with nanotube volume fraction for each of three contact pressures.

lower than previously reported optimums. In general, the optimum depends on the effect of the solid component on the conformability of the paste, and on the inherent conformability of the liquid component. The results suggest that carbon nanotube has a greater tendency to cause the conformability to decrease than carbon black or boron nitride. The trend is because of the required compromise between thermal conductivity and conformability. The conductivity increases with increasing solid content, whereas the conformability decreases with increasing solid content.

The conductance of $20 \times 10^4$ W m$^{-2}$ K$^{-1}$, as achieved by use of the paste with 0.6 vol.% nanotubes, is as high as that for solder and is above the vales for boron nitride paste based on lithium-doped PEG ($19 \times 10^4$ W m$^{-2}$ K$^{-1}$),[36] polydimethylsiloxane ($11 \times 10^4$ W m$^{-2}$ K$^{-1}$),[59] and sodium silicate ($18 \times 10^4$ W m$^{-2}$ K$^{-1}$),[59] as shown in Table I.

The limited effectiveness of solder occurs despite the high thermal conductivity of solder. This is partly because of the reaction between solder and the copper disks. This reaction results in copper–tin intermetallic compounds at the solder–copper interface.[67–69] This compound formation causes the solder not to wet the copper surface,[70] which makes it more difficult for the solder to conform to the surface topography of the copper. Conformability and spreadability can be more important than thermal conductivity in governing the performance of a thermal interface material of small thickness, as discussed in the Introduction.

Although the conductance achieved by use of the paste containing 0.6 vol.% nanotubes was comparable with that achieved by use of that containing 18 vol.% BN particles,[36] the nanotube volume fraction was much lower than the BN volume fraction. This implies that the nanotubes are much more effective than the BN particles as a thermally conductive additive for a thermal paste.

A PEG-based paste containing 3.0 vol.% ethyl cellulose and 1.25 vol.% carbon black resulted in a conductance of $30 \times 10^4$ W m$^{-2}$ K$^{-1}$,[37] and a butyl-ether-based paste containing 40 vol.% ethyl cellulose and 0.20 vol.% carbon black gave a conductance of $27 \times 10^4$ W m$^{-2}$ K$^{-1}$.[37] Thus, carbon black pastes are superior to nanotube pastes in their effectiveness as thermal pastes, despite the large particle size (30 nm) and even larger agglomerate size of carbon black. The high effectiveness of carbon black is because of the compressibility of a carbon black agglomerate[71,72] and the consequent enhanced conformability and spreadability of the paste. Carbon black has the additional advantage of its low cost compared with nanotubes, although the small size of the nanotubes may make the nanotube pastes more suitable for mating surfaces that are much smoother than those used in this work.

The thermal contact conductance values reported in this paper for pastes and solder as thermal interface materials were all obtained using the same specimen configuration, the same testing method, and the same data-analysis algorithm. The values are, therefore, particularly reliable on a relative scale. In contrast, comparison of data from different sources is less reliable, because of differences between specimen configurations and testing methods used by different researchers.

The carbon nanotube thermal paste of this work is more effective than previously reported carbon nanotube thermal interface materials.[24–30] With a nanotube content of only 0.6 vol.% this work provides a thermal paste with a much higher thermal contact conductance ($20 \times 10^4$ W m$^{-2}$ K$^{-1}$) than the value of $4 \times 10^4$ W m$^{-2}$ K$^{-1}$ (with the composition and roughness of the mating surfaces not described) achieved in previous work involving a thermal paste with a nanotube content of 3 phrs (parts per hundred resin) and a liquid crystal matrix.[24] The poor performance of the nanotube thermal paste in previous work[24] is probably because of the high nanotube content and the consequent low conformability. Although a high nanotube content (particularly one above the percolation threshold) increases the thermal conductivity within the thermal paste (particularly that in the plane of the thermal interface), it reduces the conformability. The conductance performance of the nanotube polydimethylsiloxane oil thermal paste of previous work[25] was not reported. For the nanotube array coatings in previous work, the thermal contact conductance values were $1 \times 10^4$ W m$^{-2}$ K$^{-1}$,[26] $0.2 \times 10^4$ W m$^{-2}$ K$^{-1}$,[28] $4 \times 10^4$ W m$^{-2}$ K$^{-1}$,[29] and $1 \times 10^4$ W m$^{-2}$ K$^{-1}$,[30] all of which are much lower than the value of $20 \times 10^4$ W m$^{-2}$ K$^{-1}$ for the nanotube thermal paste of this work.

## CONCLUSIONS

A thermal paste as effective as solder for use as a thermal interface material was obtained by use of single-walled carbon nanotubes (0.6 vol.%), ethyl cellulose (2.5 vol.%), and poly(ethylene glycol) dimethyl ether (2.9 vol.%) in a PEG-based dispersion.

With all the ingredients except the nanotubes the thermal contact conductance, as measured between copper mating surfaces, was $9.4 \times 10^4$ W m$^{-2}$ K$^{-1}$. In the presence of the nanotubes, the conductance was increased to $20 \times 10^4$ W m$^{-2}$ K$^{-1}$. An excessive amount of nanotubes (for example 1.8 vol.%) caused the conductance to drop, because of reduced conformability of the paste (despite the increased thermal conductivity of the paste). The corresponding conductance for solder as the thermal interface material was $20 \times 10^4$ W m$^{-2}$ K$^{-1}$. The nanotubes were more effective than hexagonal boron nitride particles, but were less effective than carbon black, for which the contact conductance was $30 \times 10^4$ W m$^{-2}$ K$^{-1}$.

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