Power Cycling and Reliability Testing of Epoxy-Based Graphene Thermal Interface Materials

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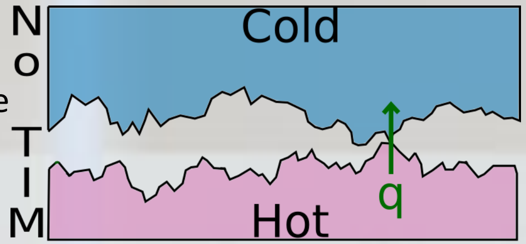
phonon optimized engineered materials

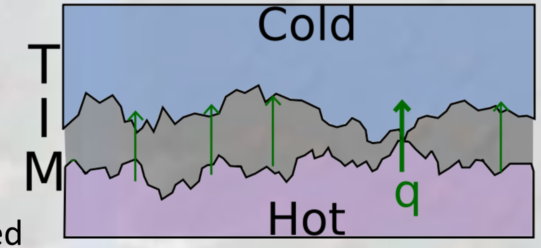
Abstract

The performance and reliability of epoxy-graphene composites have been observed over 500 power cycling measurements. These composites were prepared using an epoxy resin base and randomly oriented fillers composed of few-layer and single-layer graphene. Thermal characteristics of these composites were measured using the “Laser Flash” technique. Rather than performance degradation, the samples exhibit improvement in thermal conductivity and thermal diffusivity. Enhancement values of 15% to 25% were observed. These results suggest that epoxy-based thermal interface materials undergo a little-studied performance enhancement which may be important for the future development of thermal interface materials.

TIM Overview

- As transistors become smaller they gain efficiency, but increased device density more than compensates to increase waste heat production
- Large waste heat increases operating temperature, decreasing the lifespan of the device
- High operating temperature also causes more electrons to enter the conduction band, altering device characteristics if outside of temperature tolerances.
- Contacting solids in a heat sink have imperfect surfaces resulting in trapped air, which is an extremely poor thermal conductor

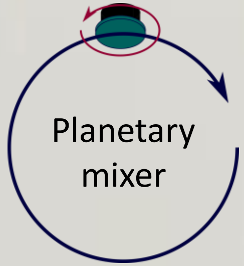




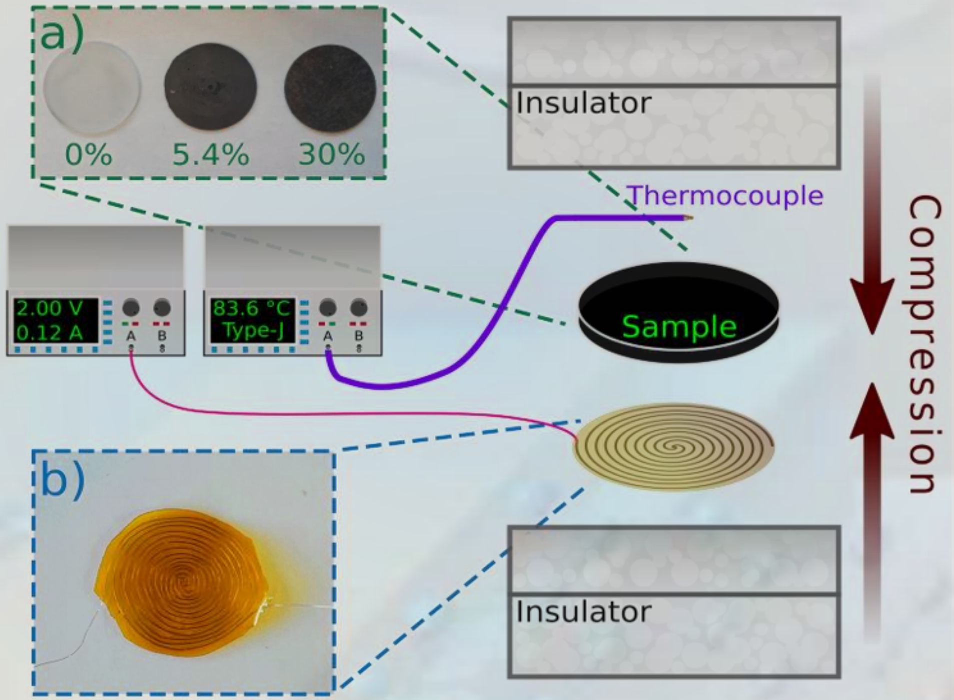
- Using a heat sink in physical contact with a semiconductor circuit is the most common solution for heat dissipation
- Due to microscopic imperfections, air gets trapped between the circuit and the heat sink, so TIMs are put in between the two surfaces to facilitate heat flow

Sample Preparation

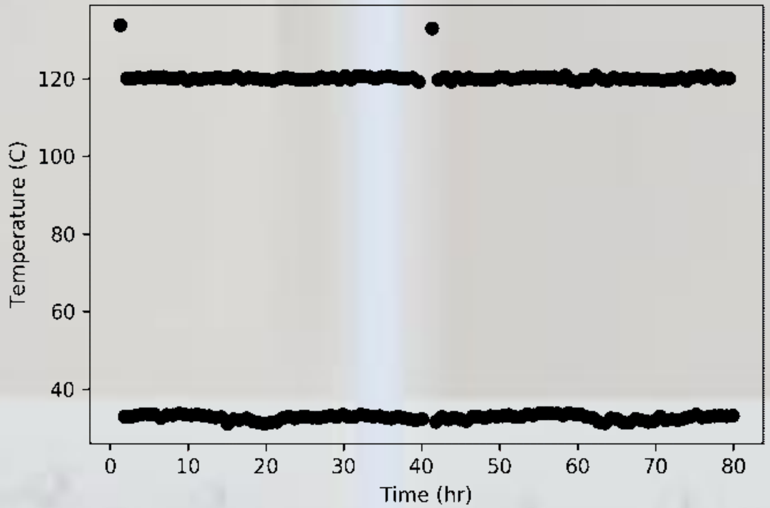
- Epoxy resin was poured into a container and put in a rough vacuum chamber for two minutes to remove air bubbles
- Container with epoxy was weighed, and this weight was used to determine the mass of other components needed
- Graphene powder was mixed into the epoxy using a bladeless planetary centrifuge for 90 seconds (1500 to 2000 RPM)
- Hardening agent was added and mixed in using the planetary centrifuge
- For graphene loading fractions of 15% weight and higher, small pressure was applied to flatten the composite
- The composites were removed from the container and polished to have a thickness between 1.5 and 1.8 mm



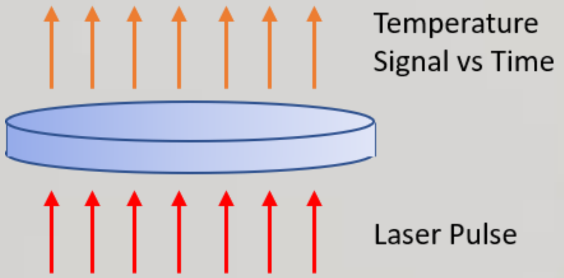
Cycling Process



- Schematic of the Cycling Setup (above)
- (a) shows the samples used in this study (percentage below each sample is the volume loading fraction of graphene used)
- Power is run through the coil in (b) to provide heat of 120 °C
- Multimeter sends temperature data to computer program for cycling control and data collection



- Figure above shows an example of the cycling process
- Temperature of the Sample vs Time
- Each dot represents one cycle (heat up, constant heat, and cool down)
- The two dots that appear higher than all the others are recalibration points



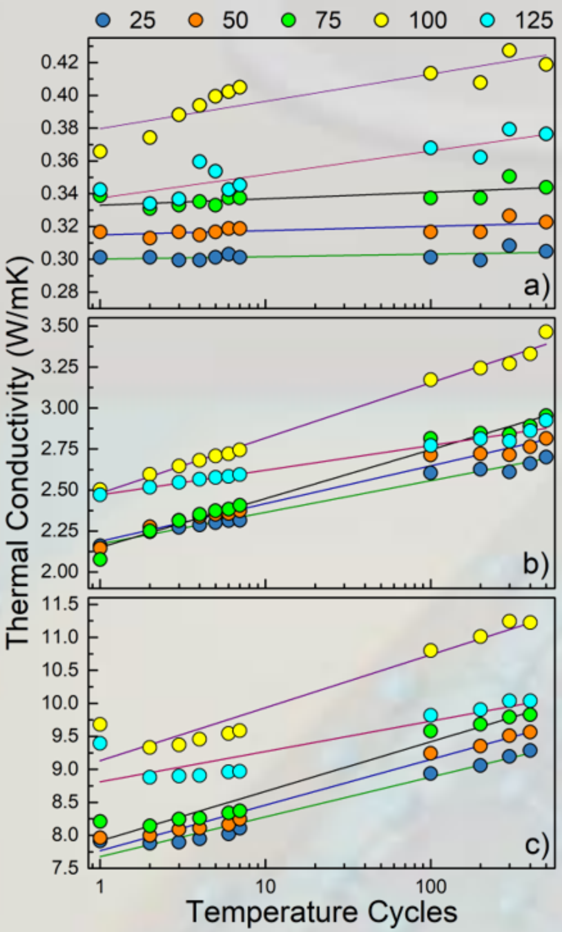
- After the prescribed cycle count, Laser Flash Analysis is used to measure thermal properties of the composites
- Laser pulse fires at sample from underneath, sample heats up, detector observes temperature change over time and calculates thermal diffusivity
- Thermal conductivity is then calculated using the equation:

$$k = \alpha \rho C_p$$

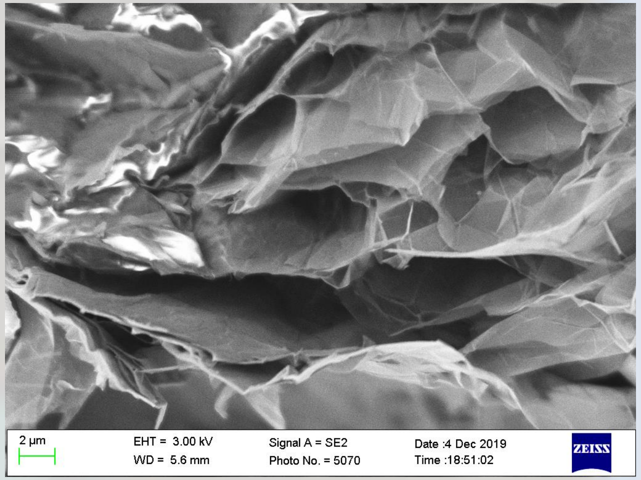
- (1) Thermal resistance equation for a TIM
- (2) Time-dependent thermal resistance equation where all parameters are measured simultaneously
- (3) Equation that represents this study: only one parameter is probed so that the resulting change is known to be dependent on a single parameter

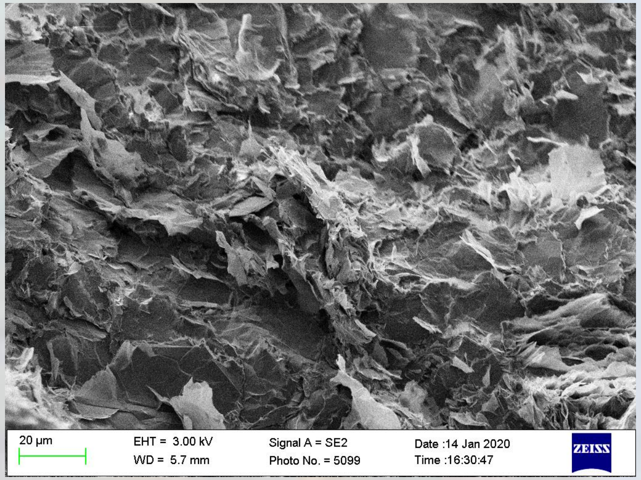
$$R_T = \frac{K}{d} + R_{C1} + R_{C2} \quad (1) \longrightarrow \quad R_T(t) = \frac{K(t)}{d(t)} + R_{C1}(t) + R_{C2}(t) \quad (2) \longrightarrow \quad R_T(t) = \frac{K(t)}{d(t)} \quad (3)$$

Results



- Thermal Conductivity versus Cycle Count
- Temperatures range from 25 °C to 125 °C
- (a) 0% vol, (b) 5.4% vol, (c) 30% vol
- All samples show improvement in thermal conductivity over increased cycle count
- Increase in conductivity as loading fraction increases was expected





- Before and After SEM Pictures of the 5.4% vol composite
- Top is before cycling, Bottom is after cycling
- Wanted to observe structural variation between before and after, but no differences were observed

Conclusion

Enhancements in thermal conductivity and diffusivity of up to 25% are observed in the composites over the course of power cycling treatments. The pure epoxy shows an improvement of only up to 7.7% at high temperatures (≥100°C). The more pronounced enhancement of thermal conductivity in graphene-loaded composites even at room temperature is attributed to increased epoxy cross-linking over the course of time spent at elevated temperatures. At higher temperatures, the epoxy polymer will increasingly cure together, more tightly gripping the graphene fillers and resulting in an enhancement of thermal coupling between graphene and polymer matrix.

Read more:

Lewis, J.S.; Perrier, T.; Mohammadzadeh, A.; Kargar, F.; Balandin, A.A. Power Cycling and Reliability Testing of Epoxy-Based Graphene Thermal Interface Materials. *C* **2020**, *6*, 26.