CARBON NANOSTRUCTURES AS THERMAL INTERFACE MATERIALS:
PROCESSING AND PROPERTIES

Thesis
Submitted to
The School of Engineering of the
UNIVERSITY OF DAYTON

In Partial Fulfillment of the Requirements for
The Degree
Master of Science in Aerospace Engineering

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May, 2011
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ABSTRACT

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The power density of electronic packages has substantially increased. The thermal interface resistance involves more than 50% of the total thermal resistance in current high-power packages. The portion of the thermal budget spent on interface resistance is growing because die-level power dissipation densities are projected to exceed 100 W/cm² in near future. There is an urgent need for advanced thermal interface materials (TIMs) that would achieve order-of-magnitude improvement in performance.

Carbon nanotubes and nanofibers have received significant attention in the past because of its small diameter and high thermal conductivity. The present study is intended to overcome the shortcomings of commercially used thermal interface materials by introducing a compliant material which would conform to the mating surfaces and operate at higher temperatures.
Thin film “labeled buckypaper” of CNF based Materials was processed and optimized. An experimental setup was designed to test processed materials in terms of thermal impedance as a function of load and materials density, thickness and thermal conductivity. Results show that the thermal impedance decreased in conjunction with the increasing heat-treatment temperature of CNFs. TIM using heat treated CNF showed a significant decrement of 54% in thermal impedance. Numerical simulations confirmed the validity of the experimental model. A parametric study was carried out which showed significant decrement in the thermal resistance with the decrease in TIM thickness.

A transient spike power was carried out using two conditions; uniform heat pulse of 24 Watts, and power spikes of 24-96 Watts. The results show that heat treated CNF was 12% more temperature resistant than direct contact with more than 50% enhancement in heat transport across it.
ACKNOWLEDGEMENTS

I am very grateful to Allah (God) for blessing me throughout this project and my entire life. This accomplishment would not have been possible without His help and desire.

It is my pleasure to thank those who made this research possible. I am heartily thankful to my advisor, Khalid Lafdi, for his guidance, encouragement and support from the initial to the final level of this research. I am grateful to him for being so kind and patient throughout this project. I would like to extend my sincere thanks to the committee members Dr. Kevin Hallinan, Dr. Lawrance Flach and Dr. Don Klosterman, for evaluating this research.

I would like to thank my collaborators at the University of Dayton Research Institute (UDRI), Dr. Lee, Alex and Matt Boehle for their tremendous support and guidance at each step of this project.

I owe my deepest gratitude to; my grandparents for their uncountable prayers for my health and success, my parents, for their incomparable love and support, my dear siblings Henna, Ali and Sana for being extremely patient and morally supportive, and last but not least, my immediate relatives for taking care of me at every single step throughout this endeavor. This work would not have been possible without the love and support of my entire family.
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Chapter I

Introduction

Thermal management is an important design consideration for number of engineered device components and packages. The need for integrating complex functions in a single circuit, together with a demand for thinner, light weight and most efficient products has been massively growing. High power and faster speeds are the major requirements of the global industry which result in excessive heating of the systems. The modern electronic systems are therefore characterized by increased density of circuits. As the circuit becomes denser, high power is required and hence high heat is generated throughout the system which should be dissipated. Rising amount of power has to be offset by efficient cooling in order for a system to be efficient. A major challenge in the field is, therefore, the ability to manage the heat without compromising on the performance of the system.

As component powers continue to grow, so do their cooling requirements. One rule of thumb says that for every 10°C rise of the junction temperature, the failure rate doubles. Thus, there is an urgent need to remove heat from current electronic devices to the surrounding air stream. Most systems are designed to
minimize thermal resistance and maximize power dissipation. But with increased miniaturization of systems and increased circuit density, today’s electronics are vulnerable and tend to generate substantial amount of heat. If the heat is not dissipated, the lifetime and reliability of the electronics will be at great risk. This is a problem that requires external thermal solutions, including heat sinks, fans, heat exchanger etc. Although these solutions are increasingly used to keep the temperature of the devices at a minimum, but materials also play a critical role. For any system assembler, when two machined surfaces are in contact, there are only few contact points between surfaces. The insulating air gaps created by non contacting areas become a thermal barrier. Current technique to overcome this barrier is either by eliminating the air gaps through an increased surface wetability (using fluid based media) or to enhance the conduction process by using highly conductive material as a thermal interface material (TIM). TIMs are used to couple these devices and interfaces with lids and heat spreaders [1-4]. Yovanovich et al. [1] calculated that replacing air with grease can reduce the thermal resistance by a factor of five or so (depending on the surfaces and contact pressure).

Thermal interface materials are commonly in the form of paste (thermal paste) [5-7], polymers [8-9], flexible graphite [10-13], phase change materials [14-15], low melting alloys [16-17] and nano-structured carbon materials [18-22]. The performance of a thermal interface material depends on its conformability, thermal conductivity and thickness [11]. An important property of any TIM is its thermal conductivity. Unfilled polymers have a thermal conductivity of about 0.1
W/m.K. All modern TIMs are composites containing fillers such as aluminum oxide, aluminum nitride, boron nitride, graphite and diamond powder, silver that increase thermal conductivity up to the 2 W/m-K. Unfortunately, high thermal conductivity alone is not enough to guarantee optimal system performance. Thermal resistance obtained from a one-dimensional heat flow calculation should also be minimized. Other properties taken into account are interfacial thickness and applied forces. In addition to thermal performance, TIMs are selected based on their inherent thermal properties, ease of use in assembly and long-term stability (reliability).

Historically, greases typically yield an interface resistance of about 1 Kcm$^2$/W. They are used to eliminate microscopic air pockets. They are often characterized as “messy” and difficult to apply due to their high viscosity. A more significant concern with application is reproducibly delivering the correct amount to achieve full coverage with the appropriately thin gap. The components require mechanical clamping pressure of about 300 kPa for optimal thermal performance. Elastomeric pads are the logical extension of greases but exhibit a higher thermal resistance range 1-3 Kcm$^2$/W. They are supplied as die-cut performs in the precise shape needed for the application. Assembly is therefore very simple with these products but require high clamping pressures (~700 kPa) are needed to achieve an adequate interface. Thermal tapes were developed as a heat sink attachment method. They eliminate the need for external clamps but thermal performance is in the 1 – 4 Kcm$^2$/W resistance range and is highly dependent on surface quality. Tapes also have very limited compliance and are
generally not suitable for current most electronic packages. However, phase change materials combine the thermal performance of grease with the convenience of an elastomer pad. Phase change materials are predominantly waxes that typically melt in the 50 – 80°C range. Essentially, they are effective conductors of heat both above and below the melt point. When operating above the melt point they are not effective as an adhesive and need mechanical support, therefore they are always used with a clamp applying pressure in the 300 kPa range. Performance levels are very close to grease, in the range 0.5 – 0.7 Kcm²/W. Solder is another TIM often overlooked. It represents the ultimate, a solid metal interface with resistance < 0.05 Kcm²/W. Despite the challenges of high temperature processing, solder is used as a thermal interface where no other viable option exists. The performance of a thermal interface material depends on its conformability, thermal conductivity and thickness. However, for the interfaces that are subjected to high temperature applications, have high roughness or not perfectly aligned, a compliant TIM must be needed. In contrast with the conventional TIMs, compliant materials conform to the mating surfaces, resist higher temperatures and do not leak out of the interface in complex applications.

Researchers are constantly seeking “better” thermal interface products. This is usually expressed as a request for higher thermal conductivity and lower thermal impedance. But is that really possible? The current study attempts to use a nanotechnology approach to look for alternative solutions.
In the light of above mentioned issues in heat management, the present study is focused on minimizing thermal contact resistance using carbon nanofiber as thermal interface materials (TIM). The performance of these TIMs was explored in pulsed heat load conditions. Chapter 2 gives a comprehensive literature review on possible approaches to overcome thermal management issues. The thermal interface material fabrication process test results and numerical simulation of the test model in terms of a parametric study on thermal conductivity and thickness of the TIMs are described in chapter 3. Chapter 4 provides TIM behavior under transient spike powers. Finally, chapter 5 concludes the present study and its implications on the ongoing and future electronics applications.
Chapter II

Literature Review

2.1 Introduction

The need for integrating complex functions in a single circuit, together with a demand for thinner, light weight and most efficient products has been massively growing. The rapid increase in the study of controlling of matter at nano-scale is a driving force in consumer applications [1-2]. Heat dissipation has become one of the most critical problems that limit improvement in performance and reliability because no two surfaces can ever make a perfect contact due to machining limitations. Tiny air gaps always remain which provide poor transmission of heat through [3]. However within the contact points, at relatively high contact pressures, hot spots may occur at the contact interface. The rapid temperature rise caused by such intensive thermal energy dissipation may be sufficiently large to cause some damage to the system [4]. Improved functionality and efficient performance of the products require more heat that must be dissipated. Heat sinks like mechanical fans, coolers have traditionally been used to transfer heat to the ambient. This solution however has been a limiting factor in designing complex and most efficient systems.
The thermal contact resistance varies considerably depending on the interface geometry, the thermal and mechanical properties of the contacting materials and the interstitial fluid [23]. The contact peak criterion is critical to the contact peak characteristics which are required to evaluate the number and size of the contact points as well as air gaps. The thermal contact conductance is the sum of the conductance of several discrete spots that exist on the interface. The thermal contact resistance can hence be determined as a function of surface parameters, material properties and contact pressure [24]. Based on published studies, both system and material approaches were considered to overcome thermal management issues [6-12].

2.2 System Approach

The system approach seems very complex and limited because of material casting issues. For solids of high thermal conductivity, the contact resistance may be reduced by increasing the area of contact spots, accomplished either by increasing contact pressure which will ‘flatten’ the peaks of the micro-roughness, and deflecting the mating surfaces to reduce any non-flatness, or reducing the roughness of the surfaces before the interface is formed by grinding the surfaces to remove non-flatness and buffing the surface to reduce micro-roughness [34]. Even by reducing the roughness through polishing the contact surfaces, it is impossible to obtain a perfect contact. Machining limitations and cost constraints are major aspects of this shortcoming. Adequate increase in the contact pressure is impractical due to the load requirements of the system.
Load constraints on electronic components and circuit boards make it unfeasible to use high contact pressure [35]. A finite distance below the heat source is required for the heat flux to become uniform. Similarly, the type and location of heat sinks also have significant effects on the overall performance of the device. There have been a number of comprehensive reviews that explored various types of heat sinks designed for efficient heat dissipation [36-49]. Lelea [36] presented a geometric optimization of the micro-heat sink with straight circular micro-channels. The inlet cross-section of rectangular shape was positioned tangentially to the tube axis with four different geometries. The results of thermal performances of heat sink were presented in terms of the temperature distribution along the bottom wall at constant heat flux and temperature of 100 W/cm² and 293 K respectively. The author observed that the lowest temperature was obtained for case 3 where the inlet channel width covered half of the tubes cross-section with minimum temperature of 313.2 K and ΔT of 10.17 K. The author reported that the results from tangential micro-heat sink showed better thermal performance than the conventional micro-heat sink with lateral inlet/outlet cross-section. Elshafei [37] performed experiments on natural convection heat transfer from circular pin fin heat sinks subject to the influence of its geometry, heat flux and orientation. The author found that the solid pin fin heat sink showed competitive performance for upward and sideward orientations. Higher heat transfer heat transfer coefficients were obtained in comparison to those of perforated/hollow pin fin ones in both arrangements. The augmentation factor was found to approximately 1.05-1.11. According to the author, the temperature
difference between the base plate and surrounding air, at the same heat input value, was found to be less for hollow/perforated pin fin heat sink than that for solid pin heat sink. Barba et al [38] evaluated the temperature distribution and thermal resistance of a polymeric heat sink with circular micro-channels. The bottom surface of the heat sink received a uniform heat flux, while the top surface was adiabatic. The experiments were carried out using two Nitrogen and Helium gases. The thermal conductivities and heat transfer coefficients were obtained to be 0.158 and 0.027 W/m-K and 3436 and 588 W/m²-K for helium and nitrogen respectively. The thermal resistance however was 5 °C/W for Nitrogen and 3.7 °C/W for Helium. Chiang et al. [39] used response surface methodology to identify the effects of design parameters of the pin-fin heat sink on the thermal performance. The height and diameter of pin-fin and the width of pitch between fins were considered as design parameters. The thermal resistance and pressure drop were calculated. Results show that the pin height and pin diameter were significant influential factors in minimizing the thermal resistance. Increase in these parameter values leads to the increase in the rate of removal heat capacity, hence thermal resistance value is decreased. The value of pressure drop was inversely proportional to the pin diameter and longitudinal and transverse pitch values. The authors also predicted the optimum values of thermal resistance and pressure drop by conducting confirmation experiments. Khor et al [40] investigated the importance of the effects of thermal radiation and its view factor on the thermal performance of a straight-fin heat sink. Three different models were developed to explore these effects on the convection
coefficient as well as the fin performance of the heat sink. Results show that the average convection coefficient for the case neglecting thermal radiation was largest with 30% error, followed by that with thermal radiation including view factor, and that with thermal radiation excluding view factor as the lowest with 60% error. The fin effectiveness was overrated for the cases when thermal radiation and view factor were excluded with more than 40% error. The authors concluded that it is reasonable to exclude thermal radiation under certain circumstances but to consider thermal radiation without incorporating the view factor could be resulting in more critical and detrimental errors. Kim et al. [41] suggested closed form correlations for thermal optimization of vertical plate-fin heat sinks under natural convection in a fully-developed-flow regime. Analytical solutions for velocity and temperature distributions for high channel aspect ratios, high conductivity ratios, and low Rayleigh numbers were presented using the volume averaging approach. The analytical solutions explained the explicit correlation for optimal fin thickness and optimal channel width, which minimize thermal resistance for given height, width, and length of heat sink. The authors reported that these correlations show that the optimal fin thickness is a function of height, the solid conductivity, and the fluid conductivity only and is independent of the Rayleigh number, the viscosity of the fluid, and the length of the heat sink. Huang et al [42] carried out experiments on natural convection heat transfer from square pin fin heat sinks subject to the influence of orientation. The authors tested a flat plate and seven square pin fin heat sinks with various arrangements under controlled environment. The results indicated that the upward and
sideward facing orientations were of comparable magnitude and showed competitive nature. Another significant factor highlighted by the authors was the porosity of the heat sink which had secondary effect on the performance of the pin fin. The optimal porosity of the heat sink was found to be around 83% for upward arrangement and 91% for the sideward arrangement. The augmentation factor (heat transfer of a heat sink relative to the base plate) was found to be around 1.1-2.5 for the upward arrangement and between 0.8–1.8 for the sideward arrangement. Jouhara and Axcell [43] described the thermal conditions within a heat sink with rectangular fins under forced convection cooling. The authors show a numerical study on key parameters of heat transfer that change with axial distance. The heat transfer coefficient and pin efficiency rapidly changed near leading edges of cooling fins. The authors reported that despite the rapid changes in these parameters, good engineering accuracy for heat sink performance is attainable using analytical methods which incorporate average values of heat transfer coefficient and fin efficiency. Lorenzini et al. [44] presented a modular heat sink consisting of elements with wavy fin profile which can be pressed together to construct the component. The temperature distribution and global thermal resistance were measured under steady state conditions. The experiments were carried out on both uniform and non uniform heat flux. The temperatures were measured by varying the location of the dissipating heaters, the power, and the number of spacers (one and five) between fan and sink. The thermal resistance is almost unaffected by the value of the ambient temperature [45]. Results show that the device performed better
when the heat source is distributed over the whole surface of the heat sink and at either side of it. Although the thermal resistances with one and five spaces were almost equal, 0.087 and 0.089 K/W respectively, the overall temperature distribution was better for single spacer compared to several spaces [44]. Ismail et al. [45] used four different types of heat sinks; Pentiums III and IV, AMD Athlon and Duron heat sinks; in order to analyze their performance. The simulation and experimental investigations were made at different Reynolds numbers. The experiments were carried out by using the air chamber with nozzle at different Reynolds numbers. The authors reported that the total surface area and fin spacing significantly affects the heat sink performance. To obtain maximum performance i.e. minimum thermal impedance, the surface should be higher with suitable fin density. Although the surface area of all heat sinks was equal, the Pentium IV heat sink showed highest performance in heat dissipation with thermal impedance of 0.4 ºC/W compared to other heat sinks. This was achieved due to larger fin spacing. Higher pressure drop across closer fins produced poor flows to pass through the fins and hence reduces the performance of other heat sinks. Wang [47] investigated the thermal performance of heat sinks with one and two pairs of embedded heat pipes. The embedded heat pipes transfer the total heat capacity from the heat source to the base plate and disperse heat into the ambient. The author found that that two and four heat pipes embedded in the base plate carry 36% and 48% of the total dissipated heat respectively. It was also reported that when the total heating power of the heat sink with two embedded heat pipes was 140 W, the total thermal resistance reached the
minimum value of 0.27 °C/W, while for the heat sink with four embedded heat pipes, when the total heating power was between 40 W and 240 W, the total thermal resistance was calculated to be 0.24 °C/W.

In the light of above cited literature, the performance of heat sinks largely depends on its geometry, the placement of the heat source and the amount of power dissipated. By the same token, heat sink’s performance varies with smooth and wavy channels because of the difference in pitch.

2.3 Materials Approach

An important way to alleviate the overheating problem is to improve the thermal contact between the heat source and heat sink by using thermal interface materials (TIMs) on the interface of contacting surfaces [48-50]. Any interstitial substance that fills the gap between contacting surfaces, and whose thermal conductivity exceeds that of air, will decrease the contact resistance [51]. The characteristics of a TIM would be high thermal conductivity, minimal thickness, would not leak out of the interface, maintains performance indefinitely, Non-toxic and manufacturing friendly (easy to apply and remove) [52]. A visual representation of interface micro-voids filled with air and TIM is shown in figure 2.1.
2.3.1 Thermal Interface Materials

Thermal interface materials (TIMs) are used to eliminate air gaps between contacting surfaces in order to maximize thermal transport. These materials are highly conductive and provide efficient heat path by conforming to the mating surfaces. Gwinn and Webb [52] presented a classification of high performance TIMs used to minimize the thermal resistance between the CPU and the heat sink, and discussed their advantages and disadvantages.

Conventional Thermal Interface materials

The most commonly used method to reduce thermal contact resistance is to introduce a thin layer of oil or grease at the interface of contacting surfaces. Mineral oil is one of the most common thermal fluids which is highly conformable and spreadable, but has low thermal conductivity. Thermal greases are composed of thermally conductive fillers dispersed in silicone or a hydrocarbon oil to form a paste. Thermal greases require no post-dispense processing and
have higher effective thermal conductivity compared to other classes of materials [53]. Greases have been used very successfully in combination with various packaging form-factors and have shown excellent performance. Corrugated copper substrate with thermal grease is described by Tzeng [54] which has high lateral conductivity and distributes the heat in the lateral direction more evenly. This would however be of value only if the heat flux were non-uniform in the interface region. Salerno et al. [55] measured thermal contact conductance of pressed contacts using Apiezon-N grease at the interface. The measurements were taken over the temperature range of 1.6-6.0 K and applied forces from 22 to 670 N. The authors reported that the addition of thermal grease between the contact surfaces resulted in an improvement of thermal conductance over uncoated surfaces to approximately a factor of 3. He [56] investigated thermal transport properties of three thermal greases (G9, G1 and S78) used as thermal interface materials in microelectronic packaging. The specific heat as a function of temperature, thermal conductivity and thermal diffusivity of these materials were measured. The author reported that among all three greases, G1 with more than 90 wt.% of highly conductive fillers, showed a thermal conductivity of 5 W/m K. Lallemand and Sartre [57] measured thermal contact resistance using several commercial greases and foils as a function of heat transfer rate and material thickness. The contact resistance decreased as the heat flux or the thickness of the material decreased. According to the authors, the highest dimensionless contact conductance factors (E) were achieved with greases (3 < E < 6). Phase change material coated foils exhibit E-values ranging from 2.5 to
3.5. Graphite or metallic foils have E-values lower than 2 and for silicone foils E is significantly reduced to less than 1. Viswanath et al. [53] presented a detailed stress analysis on a number of interface materials including thermal greases. The authors found various phenomenon associated with thermal greases under stress like 1) power cycling (loss of material due to a relative motion between the die and the base of a heat sink. The authors indicate that the assembly between 0 and 100 ºC over 7500 cycles results in a four to six fold increase in thermal resistance compared to a negligible increase in resistance for a 0 to 80 ºC exposure over 2500 power cycles. 2) thermal bake (under high-temperature bake, the formulation chemistries utilized in typical thermal greases and cause separation of the polymer and filler matrix which results in poor wetability at the interfaces, 3) Mechanical Shock and Vibration: Data collected on mechanical shock and vibration of heat-sink masses between 200-250 grams indicate that the retention of the heat sink to the processor is critical to prevent mechanical damage to the die surface. Maguire et al. [5] undertook a comprehensive study understand the thermal conditions within high power, radio frequency signal amplifiers. Experiments were carried out to determine the interface thermal resistance of a thermal grease compound and comparison of results with other alternatives. The authors reported that at maximum power dissipation, interface temperature (which accounts for 23.4% of total temperature rise) was reduced by up to 20 ºC through the use of high conductivity thermal grease. The optimum interstitial thickness depends on the thermal conductivity of the grease, which can be improved by adding metallic particles to the silicone greases [58]. Chung
[59] explored thermal contact conductance values for a number of greases and the influence of various additives to those. The author found that PEG polymer based paste gives much higher contact conductance ($11.0 \times 10^4 \text{ W/m}^2 \text{ °C}$) than silicone paste ($3.08 \times 10^4 \text{ W/m}^2 \text{ °C}$), due to its relatively low viscosity. The addition of Li salt (1.5 wt.%) to PEG raised the conductance to $12.3 \times 10^4 \text{ W/m}^2 \text{ °C}$. Further addition of water increased the conductance to $16.0 \times 10^4 \text{ W/m}^2 \text{ °C}$ and decreases the viscosity. Further addition of BN particles (18.0 vol.%) enhanced the conductance to $18.9 \times 10^4 \text{ W/m}^2 \text{ °C}$. Sodium silicate based pastes were found to give higher thermal contact conductance than polymer based pastes and oils, due to their fluidity and the consequent greater conformability [7]. A gel-like TIM has been developed by Thermoset of Lord Chemical Products [60], named as gelease, that has thermal performance similar to high thermal conductivity greases, but does not leak out of the interface. Carbon black filler [9, 61], is relatively more conformable to the contacting surfaces because of its high thermal conductivity. One added advantage of carbon is its porosity which allows penetration of the organic material into a carbon particle and hence marks it superior over conventional interface material. In a recent study [62], Hu and Chung used polyol-ester based carbon black pastes for graphite coating and/or penetration to increase the thermal contact conductance of flexible graphite between copper surfaces. The authors reported that the paste penetration by up to an effective thickness of 5 μm increased the conductance by up to 350%, 98% and 36% for thicknesses of 50, 130 and 300 μm, respectively. Whereas the paste coating up to 10 μm increases the conductance by up to 200%, 120% and 65%
for thicknesses of 50, 130 and 300 μm, respectively. Since the paste penetration effectively reduced the thermal resistivity compared to coating, the highest thermal contact conductance was found to be $1.4 \times 10^5$ W/m$^2$K by paste-penetrated flexible graphite of thickness 26 μm. Conversely, thermal greases are not manufacturing friendly as are messy, difficult to apply and remove, and can dry out with time resulting in increased thermal resistance [52]. Excess grease that flows out of the interface enables contamination and electrical shorts which limits its overall thermal performance.

In order to overcome the limitations of thermal greases, thermal pads were introduced which are composed of silicone gel in combination with a thermal medium. These pads have an inherent tack that aids placement during assembly, are more stable than phase change products, and have higher operating temperatures [63]. Due to this, thermal pads exhibit slightly better performance than greases. The Bergquist Inc. [64], one of the primary thermal interface material producers, has introduced a thermal pad named “GAP-PAD”. These pads can suit different application sectors hence the thermal conductivity could vary from 0.6 to 5 W/mK and thickness from 0.254 to 6.35 mm. Chomerics [65] produced a thermal pad named THERM-A-GAP which can offer thermal conductivity of up to 6.5 W/mK and thickness of 1 mm. Use of thermal pads could save time by speeding up the assembly process but they are too expensive to use.

Phase Change Materials (PCMs), because of its high storage capacity; have received considerable attention in recent years [66-68]. PCMs can be
classified as organic and inorganic compounds [69]. Organic PCMs can be further classified as paraffin and non-paraffin organics [75]. Abhat [69] reviewed various PCMs for low temperature latent heat storage in the range of 0-120 °C. The freezing and melting behaviors of these materials were investigated using various thermal analysis techniques. Himran et al. [71] reviewed a number of paraffin waxes for use as energy storage materials. Pal and Joshi [72] used paraffin wax, n-triacontane in a sealed heat sink to thermally control rising temperatures of the heat source. Clarksean et al. [73] performed a numerical analysis of a finned surface surrounded by a paraffin to investigate high heat flux levels. Liu and Chung [17] carried out a comparative study on phase change behavior of organic and inorganic PCMs (paraffin and microcrystalline respectively) with melting temperature close to room temperature. They found that paraffin wax was potentially good thermal interface material because of the negative super-cooling of −7°C, large heat of fusion (up to 142 J/g) and excellent thermal cycling stability. Paraffin also exhibited clear endothermic and exothermic DSC peaks which were not clear for microcrystalline. Incongruent melting and decomposition behavior was also seen in microcrystalline with high supercooling of 8°C or more and instable thermal cycling. Most PCMs with high energy storage density have an unacceptably low thermal conductivity. Therefore the performance of the PCMs can be enhanced by mixing the PCM with polymers and with particulate fillers of high thermal conductivity. Baker et al. [138] used two nonmetallic PCMs (octadecane and pentacosane) for heat storage with metal fins to enhance the effective thermal conductivity. The results
show high energy storage rates in the package and in PCMs as well as peak operating temperatures. Vesligaj and Amon [140] used an epoxy polymer as a phase change material for passive thermal control during time-varying workloads on electronics. They indicated that the operational performance of the electronics improved when such a passive thermal storage device was used. Krishnan and Garimella [143] carried out a transient analysis of the phase change process inside a rectangular enclosure for electronics cooling applications with pulsed power dissipation. The experimental investigation included the melting of a pure PCM, n-eicosane, inside a rectangular aluminum container with multiple discrete heat sources mounted on one side. A copper heat spreader was used in conjunction with the heat sources. The influence of changes in the frequency of pulses, heat source location and aspect ratio of the containment volume on the thermal performance of the PCM unit was studied. The performance of the PCM system was quantified by studying maximum temperature observed in the container, cumulative heat energy absorbed heat loss through the container walls and instantaneous heat absorbed. It was reported by the authors that the heat source location and container aspect ratio played a very significant role in the performance of the PCM system with pulsed heat input. Krishnan and Garimella [72] also performed a transient thermal analysis to investigate thermal control of power semiconductors using phase change materials, and to compare the performance of this approach to that of copper heat sinks. The authors concluded that a significant suppression of junction temperatures was achieved by the use of PCMs when compared with copper heat sinks. Pal and Joshi [73]
investigated transient transport characteristics of melting of PCMs from a uniformly dissipating source, with other walls adiabatic. It was observed that melting of PCM due to buoyancy driven natural convection, considerably dampened the effects of transient heat flux. It was shown that by estimating the heat transfer rate and molten volume fractions using the dimensionless numbers the amount of PCM and the surface area can be determined for thermal management of uniformly dissipating heat sources with various power dissipations. Alawadhi and Amon [74] carried out an experimental and numerical investigation of a TC unit for portable electronic devices. The experimental model was a prototype of a Technical Information Assistance, a wearable computer where the TC unit is embedded in the electronic device. The experimental model contained a TC unit, which is an aluminum enclosure, with aluminum foam impregnated with Eicosane PCM. The authors concluded that for the varying power operations, the TC unit highly reduced the heat source temperature fluctuations and its average temperature using PCM-foam system. Shaikh and Lafdi [75] used paraffin wax (with carbon nanotube additives) for composite thermal control (TC) system for thermal protection of electronics against transient pulsed power heat loads. The samples were subjected to both uniform and varying power conditions. The PCM composites due to greater energy storage and heat dissipation rate resulted in lower values for maximum junction temperatures for both uniform and varying power loads. The results also showed improvement in overall thermal performance of TC unit for protection against pulsed heat loads. Nurmawati et al. [76] used PCMs as potential thermal
interface materials by using two Ni-plated Cu heat spreaders. Thermal resistances were measured at initial thicknesses of 0.20–0.22 mm at various pressures, temperatures and powers. Since thermal resistance is directly proportional to the thickness of the sample, the minimum resistance of 0.13 °C/W was obtained at given thickness and 0.35 MPa. Gwinn and Webb [52] reported the thermal interface resistance values for several PCMs vary from 0.14 to 0.58 K cm²/W at 10 psi. The authors also indicated the improvement in PCM performance at higher contact pressures i.e. the thermal resistance decreased by 60% (from 0.14 to 0.058 K cm²/W) when the contact pressure was increased to 80 psi. In contrast to the above literature, there are a number of significant disadvantages of using PCMs. These fillers require moderate compression between surfaces which causes the TIM to flow. The poor thermal conductivity of PCMs at high heat flux levels is one of the major limitations towards their overall thermal performance.

Selection of an interface material not only influences the overall thermal resistance (Rth=Zth(0)) but the entire shape of the Nyquist plot as well. Although the ceramic has almost the same total thermal resistance as compared to the case without any interface material, it is clearly observed from Figure 2.2 that the middle frequency parts of the two plots do not coincide. The middle arc of the Nyquist plot is associated to the interface material. Hence a thermal resistance Rth interface can be associated to each of the interface materials [77].
Carbon Nanomaterial based Thermal Interface materials

Carbon nanotubes (CNTs) and Carbon nanofibers (CNFs) are new forms of carbon with outstanding electrical and thermal properties, high specific tensile strength and modulus [78-84]. These nanostructures are produced by many techniques including chemical vapor deposition (CVD) process [85-91]. Wang et al. [92] fabricated CNT-reinforced buckypapers by dispersing CNTs into water with the aid of epoxy resin. Multiple layers of these buckypapers with high CNT loading (up to 39 wt.%) were used to produce nanocomposites. The storage moduli of these nanocomposites were 429% higher compared to neat resin modulus. However, the major limitation of this material lies in its rigidness. Zaeri et al. [93] developed a mechanical model to determine the elastic properties of buckypaper composites. Various parameters were investigated including Young’s modulus, shear modulus and porosity of the buckypaper composite. The Young’s
modulus of buckypaper composite was 132.2 MPa compared to the predicted value of 148.6 MPa with the porosity of 67.5%. Hong and Tai [94] synthesized SWCNTs and MWCNTs to form freestanding buckypapers. The thermal conductivities at room temperature of SWCNT and MWCNT buckypapers were found to be 2.24 W/mK, and 0.36 W/mK, respectively. Xu and Fisher [95] synthesized CNT arrays directly on silicon wafers using plasma-enhanced CVD to enhance thermal contact conductance of the system. The system was based on calorimeter principle with one-dimensional reference bars and carried out in a high-vacuum environment with radiation shielding. Results show that dry CNT arrays produce a minimum thermal interface resistance of 19.8 mm$^2$ K/W, while the combination of a CNT array and a phase change material produces a minimum resistance of 5.2 mm$^2$ K/W. Cross et al. [96] grew CNTs on Si substrate using CVD process followed by metallization using Ti/Au. The coated CNTs were bonded to metallized substrates at 220 ºC. Measurements were carried out by photoacoustic technique which showed that at no applied pressure, the thermal resistance for bonded CNT films of 30 μm in length was 1.7 mm$^2$ KW$^{-1}$ where as that of CNTs up to 130 μm in length was 10 mm$^2$ KW$^{-1}$. It was hence suggested by the authors [96] that the interface resistance at the two bonded interfaces in the CNT samples dominates the overall resistance of the structure. Cola et al. [97] developed a contact resistance model to describe heat transfer across CNT array interfaces. The model revealed that the thermal resistances at single-CNT contacts dominate the thermal transport across CNT array interfaces. It was suggested that the total thermal resistance of CNT array interfaces can be
reduced by optimizing the array conformability and density such that the true contact area established in the interface is maximized. Wang et al. [98] fabricated a TIM by synthesizing aligned CNTs on both sides of a thin copper foil. The Laser flash and hot disk methods were used to measure the thermal conductivity of the CNT-TIMs. Results showed that a thicker copper foil substrate or CNT layer led to a lower overall thermal resistance of 8.78 mm² K/W. It was suggested that an enhancement in thermal conductivity of more than 290% could be obtained under an applied contact pressure of 0.01 MPa, as compared with two copper plates in direct contact. Shaikh et al. [1] fabricated a thin low density aligned CNT as TIM to explore the effect of thermal contact resistance in aluminum and graphite pieces. Compared to the direct contact, the thermal conductivity of CNT-TIM with aluminum surfaces was enhanced by 385% with an 80% decrease in thermal resistance. While with graphite surfaces, the thermal conductivity was increased by 362% with a 78% decrease in thermal resistance. All these data show the superiority of carbon based TIMs over conventional materials. High fabrication cost is however a significant limitation of CNT based materials.

Wang in [98] synthesized aligned carbon nanotubes (CNT) as a thermal interface material on both sides of a thin copper foil to measure the thermal conductivity of CNT-TIMs. It was seen that aligned CNT layer led to a lower overall thermal resistance. An enhancement in thermal conductivity of nearly 300% was obtained under certain contact pressures. Results also show that a thicker aligned CNT film produced even lower thermal resistance but there is a
trade-off between thickness and thermal resistance which should be considered to get best results. The effect of interface on the overall conductivity of composite is very small for short nanotubes, whereas interface has a significant effect on the overall thermal conductivity of the composite for long nanotubes [99-102]. Hence the interface thickness has got small effect on the thermal performance of a material. It has been verified that the interface material between two contacting surfaces can have a major influence on the thermal impedance and not only on the thermal resistance. It was observed that the thermal impedance plots depend not only on the applied conditions but also on the interface material. Material thermal performance will be affected by surface finish, contact pressure, and application properties.

Conversely, carbon nanofibers (CNFs), due to their high tensile strength, modulus, and relatively low cost, are drawing significant attention for their potential applications in nano-scale polymer reinforcement [103-106]. They are synthesized from pyrolysis of hydrocarbons or carbon monoxide in the gaseous state, in the presence of a catalyst [107-109]. There are a number of studies that show the composites formulated using vapor grown carbon nanofibers (VGCNFs) [67-72]. These CNFs distinguish themselves from other types of nanofibers, such as polyacrylonitrile or mesophase pitch-based carbon fiber, in its method of production, physical properties and structure [110]. Mahanta et al. [111] measured the thermal conductivity of vapor grown carbon nanofiber mats at different heat treatment temperatures and increasing volume fractions. The authors found that the in-plane thermal conductivity of the mats varied from 12
W/m-K to 157 W/m-K for volume fractions of 0.067 and 0.462 respectively. Thermal conductivities through the thickness were measured to be 0.428 W/m-K and 0.711 W/m-K. The authors reported that thermal conductivities through the thickness increased by the order of magnitude when CNF mats were heat treated to temperatures above 3000ºC. Thermal conductivities of individual CNFs at temperatures of 1100 ºC and 3000 ºC were calculated to be close to 1400 W/m-K and 1600 W/m-K respectively. Patton et al. [112] measured thermal conductivities of CNF composites in epoxy of up to 0.8 W/m-K for 40 vol.%. This increment over the neat resin value of 0.26 W/m-K was attributed to the complexity of thermal energy transfer within fibers. Lafdi and Matzek [113] demonstrated a significant increase in thermal conductivity of CNF composite from 0.2 W/m-K for epoxy resin to 2.8 W/m-K for a 20 wt%. Zhou et al. [114] infused various percentages of CNFs to epoxy matrix through sonic cavitations at room temperature. It was seen that the storage modulus steadily increased with an increasing weight percent of the fiber. The addition of 3 wt% of CNF yielded a 65% increase in the storage modulus. A stable CNF layer was catalytically grown on Ni foam by decomposing ethylene [115]. The presence of two distinct carbon layers was found; an apparently dense layer ‘C-layer’ at the carbon–Ni interface and a CNF layer on top of that. The nickel surface and the attached carbon layer had similar morphological features which provided strong adhesion of the C-layer to Ni which result in mechanical stability.

These properties can however be improved by tuning CNFs as a function of heat treatment temperatures. Tibbetts et al. [116] found that when CNFs are
heat treated at temperatures above 1500 °C, a significant rearrangement of the core morphology is occurred [117]. These CNFs have special structure with large inner and outer diameters, and a crystal imperfection. Besides the main product of CNFs, a small amount of amorphous carbon and metal particles can also be observed in as prepared materials. The performance of the carbon nanofibers CNFs were related to their degree of graphitic order. The degree of graphitization is determined by the temperature and the preservation period. When the temperature is up to 1873 K, the crystallite increases resulting in more conductive fiber [118]. Endo et al. [119] reported that there was a sharp decrease in the interlayer spacing from 0.342 to 0.337 nm after heat treatment to temperatures above 2100 °C. Heat treatment at graphitizing temperatures (> 2400 °C) is an effective technique for purifying and annealing the graphitic structure [120]. Parrott and Zeitler in [121] performed a catalytic activity test on the three CNF samples which resulted in the formation of a variety of hydrocarbon products, with styrene and benzene accounting for in excess of 96% of these in all cases. The ratio of styrene to other hydrocarbon products is shown in Figure 2.3.
2.4 Thermal Resistance Measurement

Thermal interface resistance and thermal conductivity measurements for thermally-enhanced interface materials are presented by a number of researchers in the published articles. Bolger [33] performed steady state thermal interface resistance measurements for single and multiple layers of epoxy tape adhesives with diamond, silver, aluminum and alumina fillers at high pressures between polished aluminum surfaces. Using these data, Bolger [33] calculated the effective value of thermal conductivity $k$ for each of the adhesives and developed a correlation to predict the volume fraction of the filler required value of thermal conductivity. Mirmira et al. [34] performed thermal contact resistance measurements using a steady-state test for a variety of adhesive materials,
including epoxies, cements and silicone. Certain thickness of each material was considered and experiments were carried out as a function of overall interfacial resistance of the system. Kilik et al. [35] presented thermal conductivity data for a variety of copper and aluminum filled epoxy adhesives. The authors reported that the thermal conductivity of adhesives can be measured effectively using the unsteady method based on conduction into a high conductivity body through a low conductivity surface film of adhesive. The thermal conductivity of adhesive decreased with the increasing temperature however, it increased substantially upon the addition of copper and aluminum powders [35].

According to the above cited literature [33-35], these methods provide much faster results than a steady-state test but do not give any relevant data for thermal resistance between a thermal interface material (TIM) and the interfacing surface, which is as significant as the bulk conduction resistance. In this study however, a steady state method will be presented for determining the bulk thermal conductivity and thermal contact resistance across the interface using thermal impedance measurements. These tests will be performed with a hot and cold plate technique based on the calorimeter principle [122]. The TIM sheet was sandwiched between a hot and a cold plate and the heat flow is measured [123]. The thermal resistance is then calculated as the temperature difference between the hot and cold plate divided by the heat flow. The hot and cold plates are perfectly isothermal and they make a perfect contact with the interface material under test. Zhu and Wang [30] stated that the thermal interface resistance (TIR) can be reduced significantly by exerting the pressure or spreading an adhesive
between two contacting surfaces. Thermal conductivity is an important property of composites application for electronic packaging, thermal insulation, heat spreader, etc. [124]. Voller and Tirovic [125] presented some methods for decreasing the thermal resistance across a bolted joint, such as using thin aluminum gasket at the interface. Cheng and Madhusudana [126] investigated the contact heat transfer of fin–tube exchangers where zinc, tin, silver, and gold were chosen as plating materials. The minimum TIR was obtained when the tube was coated with tin. This indicates that, although the thermal conductivity is important, the softness of the plating material also plays an important role in enhancing the thermal conductance of the interface.

2.5 Proposed Methodology

To achieve high thermal conductivity, CNFs at various heat treatment temperatures (PR24-PS, PR24-LHT, and PR24-HHT) were used [133]. These CNFs have special structure with large diameters (less than 100nm) and some crystal imperfections. The performance of the CNFs will be related to their degree of graphitizability. This study is focused on the fabrication and characterization of CNF buckypapers. The analysis is performed by varying the ratio of CNF and binder polyvinyl alcohol (PVA) and, heat treatment temperatures of CNFs. These altering parameters should lead to a buckypaper with specific density (i.e. thickness) and thermal conductivity.

There are numerous synthetic polymers that are used in fabrication of composite materials. Polymer based composites reinforced with a small percentage of strong fillers can significantly improve the mechanical, thermal,
and barrier properties of the pure polymer matrix [127]. Moreover, these improvements are achieved through conventional processing techniques without any detrimental effects on process ability, appearance, density, and ageing performance of the matrix. Many researchers synthesized micro-scale fillers with epoxy resin [128-130]. Zhou and Pervin [114] infused various percentages of CNFs to epoxy matrix through sonic cavitation at room temperature. It was seen that the storage modulus steadily increased with an increasing fiber weight percent. The addition of 3 wt% of CNF yielded a 65% increase of the storage modulus. Another most common water-soluble polymer, Polyvinyl Alcohol (PVA), was used to make composite fibers which contained large fraction of nanotubes [131]. Composite nanotube–PVA fibers exhibit an extremely high toughness, superior to that of any other materials [132-133]. They are electrically and thermally conductive and could therefore be the basis for lightweight, strong and conducting composites.
Chapter III

Material Fabrication and Characterization

3.1 Materials Fabrication

The as-received nanofibers consist of a variety of carbon configurations like helical, straight, nested, carbon blacks with narrow distribution of diameters and lengths (Figure 3.1).

Figure 3.1 Bright-field image of pristine PS carbon nanofibers.
The five major carbon constituents represented as carbon nanofibers are shown in Figure 3.2. Carbon blacks, sometimes recognized as soot, are nanometric spheres of agglomerated carbon. Helical carbon nanofibers, or nanocoils, consist of carbon nanofibers that have a configuration similar to that of a DNA strand. Straight carbon nanofibers exist as a series of coaxial carbon cylinders surrounding a central hollow tube. The bamboo species are similar to that of the straight carbon nanofibers, except that they are segmented along their length. Nested carbon nanofibers have an orientation similar to that of a set of stacked Dixie Cups with a hollow core and are also referred to as fishbone type carbon nanofibers.

1. Carbon Blacks: Minor

2. Helical: Minor

3. Straight: Major

4. Nested (D.C.): Dominant

5. Bamboo: Dominant

Figure 3.2 Models of various nanofiber configurations

Carbon at low temperatures exhibits local molecular ordering. As a carbon sample is heat treated, the increase in temperature results in the aromatic
molecules stacking into a columnar structure. Further heat treatment causes these columns to join together forming a distorted, wavy structure. Heat treatment temperatures of 2500°C or higher compress the distorted graphene layers forming an aligned structure. Graphitic carbon will attain the minimum interlayer spacing in the graphite order between graphene layers (Figure 3.3). Heat treatment of pristine carbon nanofibers to 3000°C results in a straightening of the graphene layers. The minimum interlayer spacing was attained for the HHT nanofibers.

Figure 3.3 Carbon plane structure as a function of heat-treatment temperature [134]

As shown in a TEM micrograph (Figure 3.4) the layers within the Dixie Cup carbon nanofiber coalesced following heat treatment resulting in localized ordering of the graphene layers and continuous planes. At this magnification the
inclination angle of each cup is apparent. The use of grey-scale allowed the walls of the nanofibers to appear dark due to the high electronic density in this region.

![Image]

Figure 3.4 Bright field micrograph of Dixie Cup carbon nanofiber structure

High resolution imaging of the graphene layers (Figure 3.5) indicated the alignment of the layers without disclination defects. There is no change in the inclination angle to the central core axis. The edge of any pair of graphene layers is rounded to encapsulate the exposed edge of the carbon planes, allowing the exposed graphene planes to attain a level of maximum structural stability.

Each of the pristine carbon nanofibers was tested for electrical volume resistivity measurement. The description of each of the nanofibers is provided in Table 3.1.
Figure 3.5 High-resolution imaging of localized area of Dixie Cups carbon nanofiber structure

Table 3.1 Description of tested carbon nanofibers

<table>
<thead>
<tr>
<th>ASI Sample</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>AG Grade</td>
<td>As-grown nanofiber (processing temperature 1100°C)</td>
</tr>
<tr>
<td>PS Grade</td>
<td>Pyrolytically stripped nanofiber (processing temperature 1100°C)</td>
</tr>
<tr>
<td>LHT Grade</td>
<td>Nanofiber after heat treatment temperature up to 1500°C</td>
</tr>
<tr>
<td>HHT Grade</td>
<td>Nanofiber after heat treatment temperature to 3000°C</td>
</tr>
</tbody>
</table>

The results of the volume resistivity testing are presented in Figure 3.6. There is a significant drop in the volume resistivity between the AG and PS nanofibers and between the PS and LHT nanofibers. However, the change in
the temperature difference between the AG, PS, and LHT is only 400°C. This indicated an anomaly in the observed decrease in resistivity. Further heat treatment of the nanofibers does not significantly reduce the volume resistivity. If the volume resistivity were only a function of heat treatment temperature, the values should result in a relatively linear decrease with a sharp decrease after 2500°C. There is likely an additional factor in the determination of volume resistivity which resides either in the surface contact or deposition of a dielectric material.

![Graph showing volume resistivity](image-url)

**Figure 3.6** Volume resistivity of as received carbon nanofibers at 108psi

A mixture of carbon nanofibers (CNF) and water soluble binder polyvinyl alcohol (PVA) was subjected to ultrasonic dispersion process. A wide range of
parameters can be varied in this process to optimize the thermal properties of the thermal interface material (TIM). In the present study however, the parameters were kept constant to achieve best results in terms of thickness and conformability of the TIM. In the first step, 0.8 g of PVA was mixed with 400 ml of water. Secondly, 1.2 g of CNFs was added to the mixture before being subjected to ultrasonic dispersion process for four hours. The solution was then filtered using latex filter Millipore (0.7um). Figure 3.8 shows the filtration process and the filtered sample. The sample was then heat treated at 400 ºC for 15 minutes to evaporate residual PVA and water. The final sample obtained was a CNF compliant material (buckypaper) with less than 1% impurities (Figure 3.9). Figure 3.7 shows the step wise buckypaper fabrication process.

![Fabrication process of buckypaper TIM](image)

Figure 3.7 Fabrication process of buckypaper TIM
The characterization tool used in this study is primarily based on the ASTM D5470 test method for measuring thermal resistance. Major improvements have been made to the base design (Appendix A). It measures the thermal impedance and thermal conductivity under a range of clamping forces and thicknesses, for thermal interface materials (TIMs).

The construction of the experimental setup is based on a steady-state heat flow along the sample (TIM) placed between two aluminum blocks, one attached to the heating and another to the cooling plate, as shown in Figure 3.10. Each block is one square inch in size and has two thermocouples inserted into the heat.
transport path. Power is applied to generate heat in the heating block. The material under test is placed between the upper block attached to the ‘hot surface’ and lower block attached to the ‘cold surface’. The lower block is designed utilizing a liquid cold plate which has a regulated supply of coolant of known temperature. Load is applied on top of the heating plate by the MTS Electromechanical displacement unit. Data acquisition device reads the temperatures from four thermocouples when thermal equilibrium is established, the data is then transferred to the Excel spread sheet to perform the thermal interface resistance calculations.

The control unit (Figure 3.11) consists of a power controller, a temperature controller and a data acquisition device. A 120 V ~ 60 Hz dimmer is used as a power controller and VFL series PID as a temperature controller. NI-USB 9162 data acquisition device is used to read temperature reading from four thermocouples. Electrical power is applied to the controller via a power switch.

The MTS Electromechanical displacement unit was programmed and configured using Test Works 4.0. This includes pre-specimen, specimen and post-specimen programming of the crosshead movement. Appendix A shows the detailed parameters of each step in the program.

Prepared test sample is applied to the lower aluminum block. All tests are carried out at loads of 45, 90, 135 and 180 kg. The heater at the upper block was set to 80 ºC and the chilled water bath at the lower block was set to 10 ºC. The heat generated from the heater flows down thru the metering block, thru the TIM, thru the lower metering block and into the liquid cold plate controlled by the
chilled water bath. The free convection and radiation terms were neglected since the test rig is located in a controlled atmosphere room. At steady state, four temperature readings are taken with the help of the data acquisition. The temperature difference is taken to calculate the thermal impedance at each load. The extension in the displacement cell measures the thickness of the TIM under test which is used to calculate the effective thermal conductivity of the TIM. The total heat loss from the system is estimated to be less than 3%.

Figure 3.10 Experimental Setup

Figure 3.11 Control unit
Figure 3.12 Schematic of the test setup with resistances

Figure 3.12 represents total resistances of the two contacting surfaces as well as those at the interface. The determination of the heat flux through a thermal interface material is equal to the electrical power loss. Heat either transfers through the TIM contacts or the air gaps. The remaining air conductivity and the contact conductivity (between TIM and the aluminum block) could be added to achieve global thermal conductivity $h_{\text{global,Al}}$ as shown in Eq. 1.

$$h_{\text{global,Al}} = \frac{1}{h_{\text{Al-TIM}, S1}} \left( h_{\text{Al-Air}, (S - S1)} \frac{1}{\lambda_{\text{Air}, (S - S1)}} \frac{1}{e_{\text{Air}}} + \frac{1}{h_{\text{Al-Air}, (S - S1)}} \frac{1}{\lambda_{\text{Air}, (S - S1)}} \frac{1}{h_{\text{Air-TIM}, (S - S1)}} \right)$$  \hspace{1cm} (Eq. 1)
Where;

\[ h_{\text{global, Al}} = \text{Global thermal contact conductivity (W/m}^2\text{-K)}; \]

\[ h_{\text{Al-TIM}} = \text{Thermal contact conductivity between Al and the TIM. (W/m}^2\text{-K)}; \]

\[ h_{\text{Al-Air}} = \text{Thermal contact conductivity between Al and Air (W/m}^2\text{-K)}; \]

\[ \lambda_{\text{TIM}} = \text{Thermal conductivity through the TIM sheet (W/m-K)}; \]

\[ e_{\text{Air}} = \text{Diameter of the air gap (mm)}; \]

\[ S = \text{Sample area (mm}^2); \]

\[ S1 = \text{Upper Aluminum block/TIM contact surface (mm}^2); \]

\[ S2 = \text{Lower Aluminum surface/TIM contact surface (mm}^2). \]

The global resistivity of the system is represented in figure 3.13,
Figure 3.13 Global resistivity of the system
With the addition of a TIM at the interface, the resistance at each sub-interface is added to the global resistivity of the system (Figure 3.13). The final schematic of an equivalent thermal circuit with a TIM inserted at the interface (Figure 3.14)

Figure 3.14 Thermal resistances at each component

The global heat flow $\varphi$ is measured by the temperature difference between two thermocouples of the lower aluminum block (physically known test).
\[ \varphi = \frac{(T_1 - T_2) \cdot S}{R_{Al2}} \quad (Eq. 2) \]

And \( S = 1 \text{ in}^2 \) (contact surface), hence;

\[ R_{Al2} = \frac{e_2}{\lambda_{Al}} = \frac{y(T_1) - y(T_2)}{\lambda_{Al}} \quad (Eq. 3) \]

To determine the thermal resistivity of the TIM, the temperatures must be calculated between the interfaces \( T_{int1} \) and \( T_{int2} \), determined by the four thermocouples:

\[ T_{int1} = T_3 - \frac{T_4 - T_3}{2} \quad (Eq. 4) \]

\[ T_{int2} = T_2 + \frac{T_2 - T_1}{2} \quad (Eq. 5) \]

Hence, the thermal resistivity of the TIM is defined by:

\[ R_{TIM} = \frac{(T_{int1} - T_{int2}) \cdot S}{\varphi} \quad (Eq. 6) \]

The global thermal impedance at the interface can be calculated as a function of thermal parameters:
This law of the thermal resistivity function of the thickness \( e \) is determined by a numerical linear fit as shown in Figure 3.15.

\[
R_{TIM}(e) = A \cdot e + B = \frac{e}{\lambda_{TIM}} + \frac{2}{h_{TIM-Al}} \quad (Eq. 7)
\]

After individually calibrating each ‘T’ type thermocouple using chilled water bath, each thermocouple reading is stored by using the LabView software. In order to determine the steady state condition, temperature curves are plotted as a function of time (Figure 3.16)
Figure 3.16 Calibration of the system

TestWorks 4.1, MTS software, was programmed to control the Electromagnetic Load Cell, was calibrated by using the ‘Proportional Integral Derivate’ (PID) parameters. It was very important to maintain a good proportion between two aluminum blocks in order for crosshead to balance the load for a given time before returning to the initial position (Figure 3.17). Table 1 shows the values of $K_p$, $K_i$ and $K_d$, at which the crosshead was stabilized. The maximum load to be applied was set to 180 kg (400 lbf) so as not to damage the load cell and work pieces.

Table 3.2 Configuration parameters of MTS Displacement Unit

<table>
<thead>
<tr>
<th>PID Parameters</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>$K_p$</td>
<td>0.000100</td>
</tr>
<tr>
<td>$K_i$</td>
<td>0.000010</td>
</tr>
<tr>
<td>Max Integral (in/min)</td>
<td>2.362200</td>
</tr>
<tr>
<td>Derivative Interval</td>
<td>2.000000</td>
</tr>
<tr>
<td>Maximum Speed (in/min)</td>
<td>23.62200000</td>
</tr>
</tbody>
</table>
After every test, the aluminum blocks surfaces are cleaned up by Acetone to remove any residual left. It is very essential to maintain same surface roughness during all tests to accurately compare the results. The effect of the heat transfer coefficient $h(\text{Al-Sample})$ is directly related to the roughness and the residual material.

The thermal resistivity calculation (temperature, thermocouple position and area):

$$R_{TIM} = \frac{(T_{int1} - T_{int2}) \cdot S}{\varphi} = \frac{y_{T1} - y_{T2}}{2 \cdot \lambda_{TIM}} \cdot \frac{3T_3 - T_4 - 3T_2 + T_1}{T_2 - T_1} \cdot S$$
Error \( (R_{TIM}) = \)

\[
\frac{\Delta y}{2.\lambda_{TIM}} \times \frac{3T_3 - T_4 - 3T_2 + T_1}{T_2 - T_1} \times S + \frac{y_{T1} - y_{T2}}{2.\lambda_{TIM}} \times S \times \left( \frac{3}{T_2 - T_1} - \Delta T_4 \right) \\
+ \Delta T_1 \frac{2T_1 + 2T_2 - 3T_3 + T_4}{(T_2 - T_1)^2} \times \Delta T_2 \frac{-2T_1 + 3T_3 - T_4}{(T_2 - T_1)^2} \) \times \frac{1}{R_{exp}}
\]

Where:

\( y_{T1} - y_{T2} \) = distance between two thermocouples (Error – 0.02 inch),

\( T_1, T_2, T_3, T_4 \) = Thermocouple temperatures (Error – 0.05K),

\( S \) = Contact area (mm\(^2\)),

\( R_{exp} \) = Experimental thermal resistivity.

---

**Table 3.3 Final error calculation of thermocouples**

<table>
<thead>
<tr>
<th>( Y(T1), Y(T2) )</th>
<th>( 0.500 )</th>
<th>( \Delta y )</th>
<th>( 0.02 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>( T_1 )</td>
<td>27.29</td>
<td>( \Delta T1 )</td>
<td>0.05</td>
</tr>
<tr>
<td>( T_2 )</td>
<td>31.95</td>
<td>( \Delta T2 )</td>
<td>0.05</td>
</tr>
<tr>
<td>( T_3 )</td>
<td>43.64</td>
<td>( \Delta T3 )</td>
<td>0.05</td>
</tr>
<tr>
<td>( T_4 )</td>
<td>47.87</td>
<td>( \Delta T4 )</td>
<td>0.05</td>
</tr>
<tr>
<td>( \lambda_{Al} )</td>
<td>6.02</td>
<td>( \lambda_{Al} )</td>
<td>0.05</td>
</tr>
<tr>
<td>( S )</td>
<td>1.00</td>
<td>( ERROR )</td>
<td>0.029</td>
</tr>
</tbody>
</table>
3.2 Validation of the Test Setup

First step in the process was to validate the test setup with the published results. In order to validate the designed setup, various dry tests were carried out to observe the functionality of the setup. As the next step, Arctic Silver thermal interface material was analyzed at different loads and thicknesses to compare the obtained results with the published ones (Figure 3.18).

Table 3.4 Validation of the setup

<table>
<thead>
<tr>
<th>Arctic Silver Thermal Interface Material</th>
<th>Load (kg)</th>
<th>Thickness (mm)</th>
<th>Thermal Impedance (K/W)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Published</td>
<td>135</td>
<td>0.025</td>
<td>0.0136</td>
</tr>
<tr>
<td>Experimental</td>
<td>135</td>
<td>0.025</td>
<td>0.0137</td>
</tr>
</tbody>
</table>

Figure 3.18 Thermal impedance of Arctic Silver as a function of load
The behaviour of thermal interface materials cannot be analyzed without taking into account the change in thickness of the sample. In order to determine the change in TIM thickness at different loads, the change in cross position is monitored. During the process, the crosshead position changes due to two reasons, the density of the TIM (the conformability) and due to thermal expansion of aluminium surfaces. At high temperatures, the thermal expansion of aluminium surfaces was monitored by carrying out dry tests (without any TIM). The initial and final positions of the upper aluminium block were determined at various loads. In order to analyze buckypaper thermal interface materials under various loads, the sample thickness $e$ can be determined by;

\[ e = C_{cp} - I_{cp} \]  

(Eq)

Where;

$C_{cp}$ = Current crosshead position

$I_{cp}$ = Initial crosshead position
Figure 3.19 Thickness calculations of samples by using crosshead position

In figure 3.19, four initial crosshead position lines (45, 90, 134, and 180) are shown during the direct contact calibration. Initially the residual values are approximately 1. When the TIM is inserted into the interface, the current crosshead position is 2.374 mm at 45 kg. The sum of four temperature readings is 150 ºC. Therefore the initial crosshead position for this temperature is 2.319 mm. The thickness of the sample is 0.055 inches (2.374 -2.319). The error for thickness calculation is less than 5%.
3.3 Experimental Characterization

3.3.1 Thermal conductivity calculation

An experimental characterization of heat conduction between two contacting surfaces has been developed which enables the measurement of thermal impedance as a function of load and thickness. Four buckypaper samples with different thicknesses were tested in order to find the influence of those on global thermal resistance. It was seen that as the load increases, both slope coefficient $A$ and $Y$-intercept $B$ (Eq. 7) decrease (Figure 3.20). Thus, thermal conductivity through the TIM ($\lambda_{\text{TIM}}$) and at the interface ($h_{\text{global,Al}}$) increase with the load.

![Figure 3.20 Thermal resistance of buckypaper TIMs as a function of thickness at various loads](image-url)
Table 3.5 Thermal conductivity values at various loads

<table>
<thead>
<tr>
<th>Thermal Conductivity</th>
<th>Applied Load (kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>45</td>
</tr>
<tr>
<td>Through the TIM (W/m·K) $\lambda_{\text{TIM}}$</td>
<td>0.71</td>
</tr>
<tr>
<td>At the Interface (W/m²·K) $h_{\text{global,Al}}$</td>
<td>3.99x$10^4$</td>
</tr>
</tbody>
</table>

Thermal conductivity $\lambda_{\text{TIM}}$ increases with the load due to significant decrease in TIM density. The $h_{\text{TIM-Al}}$ also increases due to better conformability of the TIM at higher loads. Figure 3.21 shows the graphical representation of the TIM in terms of $\lambda_{\text{TIM}}$ and $h_{\text{global,Al}}$.

![Figure 3.21 Plot showing increase in thermal conductivity of the sample as load increases](image)

Figure 3.21 Plot showing increase in thermal conductivity of the sample as load increases
The initial porosity of the buckypaper was approximately 0.4 but at higher loads, because of compression, carbon fibers tend to get closer allowing better heat transport. It is well known that the thermal conductivity of air is lower than that of carbon nanofibers and contact points increase with the pressure, the thermal conductance through the thermal interface material ($\lambda_{\text{TIM}}$) increases. Figure 3.22 shows the decrease in porosity with increasing load.

![Diagram showing initial and compressed samples with air gaps and load](image)

**Figure 3.22 Decrease in porosity of the sample as the load increases**

**3.3.2 Thermal Impedance comparison**

The performance of buckypaper TIM with different heat treated CNFs was investigated in comparison with the direct contact to analyze the advantage of using this compliant material. For this purpose, the TIM of approximately 0.004 inch thickness was selected. It was seen that the thermal impedance values with high heat treated CNF (HHT-CN) buckypaper were significantly better than low
heat treated (LHT) and PS (pyrolytically stripped). For the buckypaper with HHT-CNFS as compared to the direct contact between two surfaces at maximum load, the thermal impedance was reduced by 54% from 0.151 K/W to 0.069 K/W (Figure 3.23). Since the thickness of the TIM decreases with increasing load, the final bond line thickness attained was 0.075 mm at applied load of 180 kg (Table 3.7).

Table 3.6 Thickness comparison of three types of CNF buckypapers

<table>
<thead>
<tr>
<th>Load (kg)</th>
<th>Thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>CNF-HHT</td>
</tr>
<tr>
<td>45</td>
<td>0.119</td>
</tr>
<tr>
<td>90</td>
<td>0.089</td>
</tr>
<tr>
<td>135</td>
<td>0.084</td>
</tr>
<tr>
<td>180</td>
<td>0.082</td>
</tr>
</tbody>
</table>

Table 3.7 Thermal impedance comparison of direct contact and CNF TIMs

<table>
<thead>
<tr>
<th>Load (kg)</th>
<th>Thermal Impedance (K/W)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Direct Contact</td>
</tr>
<tr>
<td>45</td>
<td>0.391</td>
</tr>
<tr>
<td>90</td>
<td>0.264</td>
</tr>
<tr>
<td>135</td>
<td>0.212</td>
</tr>
<tr>
<td>180</td>
<td>0.151</td>
</tr>
</tbody>
</table>
Figure 3.23 Comparison of thermal impedance of various heat treated CNF buckypapers with the dry contact

Table 3.8 – Thermal Performance comparison of various TIMs

<table>
<thead>
<tr>
<th>Product</th>
<th>Thermal Impedance (K/W)</th>
<th>Enhancement</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dry Contact</td>
<td>0.151</td>
<td>Reference</td>
</tr>
<tr>
<td>Aremco 640</td>
<td>0.071</td>
<td>52.98%</td>
</tr>
<tr>
<td>Shin-Etsu MicroSi® G751</td>
<td>0.098</td>
<td>35.10%</td>
</tr>
<tr>
<td>Dow Corning® TC-5121</td>
<td>0.096</td>
<td>36.42%</td>
</tr>
<tr>
<td>Omegatherm 201</td>
<td>0.081</td>
<td>46.36%</td>
</tr>
<tr>
<td>PowerFilm 51</td>
<td>0.474</td>
<td>-213.91%</td>
</tr>
<tr>
<td>Thermal Grease TIC-1000A</td>
<td>0.072</td>
<td>52.32%</td>
</tr>
<tr>
<td>HighFlow 565U</td>
<td>0.221</td>
<td>-46.36%</td>
</tr>
<tr>
<td>CNF-PS</td>
<td>0.833</td>
<td>-451.66%</td>
</tr>
<tr>
<td>CNF-LHT</td>
<td>0.723</td>
<td>-378.81%</td>
</tr>
<tr>
<td>CNF-HTT</td>
<td>0.069</td>
<td>54.30%</td>
</tr>
</tbody>
</table>
Table 3.8 shows thermal performance of various commercially used TIMs in comparison with that of CNF based buckypapers. Most conventional TIMs (e.g. thermal greases) are liquid based and messy in installation. They also require flawless application to ensure uniform distribution at the interface. The applied pressure can cause the TIM to leak out of the interface. In contrast, the buckypaper TIM is dry in nature and provides competitive thermal performance.

### 3.3.3 PVA/CNF Ratio

Four samples of CNF-HTT buckypaper were prepared to analyze the ratio of binder material PVA to the CNF-HHT. By varying the PVA weight and keeping the CNF weight constant (0.5g), different thermal impedance behaviors were seen. For each composition, two samples with different thicknesses were analyzed to find the thermal impedance as a function of thickness. All compositions were finally compared at a sample thickness of 0.1 millimeter.
Figure 3.24 Thermal resistance of BP1 as a function of thickness

Figure 3.25 Thermal resistance of BP2 as a function of thickness
Figure 3.26 Thermal resistance of BP3 as a function of thickness

Figure 3.27 Thermal resistance of BP4 as a function of thickness
From above figures (3.24-3.27), it was noticed that two samples with same PVA to CNF ratio but different global weights like (BP1 and BP2) show identical thermal behavior. It is hence important to define the samples by its PVA to CNF ratio and not its global weight. However, the increase in the weight percent of PVA with constant CNF weight has minimum effect on the thermal resistance at the interface.

Hence the thermal impedance graph in terms of PVA to CNF ratio was analyzed (Figure 3.28) and it was noticed that at low pressure, as the ratio between two materials increased, the thermal impedance increased. However, the thermal impedance remains constant at high temperatures, regardless of the ratio between PVA and CNF.

Table 3.9 Thermal impedance ratio of PVA to CNF Buckypaper

<table>
<thead>
<tr>
<th>PVA/CNF Ratio</th>
<th>CNF Buckypaper</th>
<th>Thermal Impedance (K/W)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>45 kg</td>
</tr>
<tr>
<td>2</td>
<td>BP1</td>
<td>0.443</td>
</tr>
<tr>
<td>4</td>
<td>BP2</td>
<td>0.411</td>
</tr>
<tr>
<td>6</td>
<td>BP3</td>
<td>0.523</td>
</tr>
<tr>
<td>8</td>
<td>BP4</td>
<td>0.543</td>
</tr>
</tbody>
</table>
Similarly, the ratio of PVA to CNF is plotted in terms of thermal conductivity through the TIM thickness (Figure 3.29) and overall heat transfer coefficient (Figure 3.30).
Figure 3.29 Thermal conductivity comparison of PVA to CNF ratio

Figure 3.30 Heat transfer coefficient as a function of ratio of PVA to CNF
3.3.3.1 PVA Effects

In order to understand the effects of water soluble binder material Polyvinyl Alcohol (PVA), seven buckypaper samples were fabricated by varying the weight of PVA by 1 gram while keeping the CNF weight constant to 0.5 grams (Table 3.10). The thermal impedance was calculated for each sample in terms of a) PVA weight (Figure 3.31) and b) applied load (Figure 3.32).

Table 3.10 Weight ratios of various buckypaper TIMs

<table>
<thead>
<tr>
<th>Material</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>CNF (g)</td>
</tr>
<tr>
<td>BP1</td>
<td>0.5</td>
</tr>
<tr>
<td>BP2</td>
<td>0.5</td>
</tr>
<tr>
<td>BP3</td>
<td>0.5</td>
</tr>
<tr>
<td>BP4</td>
<td>0.5</td>
</tr>
<tr>
<td>BP5</td>
<td>0.5</td>
</tr>
<tr>
<td>BP6</td>
<td>0.5</td>
</tr>
<tr>
<td>BP7</td>
<td>0.5</td>
</tr>
</tbody>
</table>
Figure 3.31 Thermal impedance values as a function of PVA weight

Figure 3.32 Thermal impedance of various TIMs as a function of load
3.3.3.2 CNF Effects

To study the effects of carbon nanofibers (CNF) on the conformability of the TIM, six buckypaper samples were created by varying the CNF weight by 0.5 grams while keeping PVA weight constant to 3 grams (Table 3.11). The thermal impedance was calculated for each sample in terms of a) CNF weight (Figure 3.33) and b) applied load (Figure 3.34).

Table 3.11 Weight ratios of various buckypaper TIMs

<table>
<thead>
<tr>
<th>Material</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>CNF (g)</td>
</tr>
<tr>
<td>BP1</td>
<td>0.5</td>
</tr>
<tr>
<td>BP2</td>
<td>1.0</td>
</tr>
<tr>
<td>BP3</td>
<td>1.5</td>
</tr>
<tr>
<td>BP4</td>
<td>2.0</td>
</tr>
<tr>
<td>BP5</td>
<td>2.5</td>
</tr>
<tr>
<td>BP6</td>
<td>3.0</td>
</tr>
</tbody>
</table>
Figure 3.33 Thermal impedance values as a function of CNF weight

Figure 3.34 Thermal impedance of various buckypaper TIMs as a function of load
The main purpose of the test setup was to characterize the TIMs in terms of effect of the ratio of CNF and PVA (binder material) on the global thermal resistance, and thermal impedance as a function of load and thickness. TIMs with high heat treated fibers minimized the global resistance of the system by 54% compared to the direct contact.

### 3.4 Modeling

The Thermal Impedance test setup was modeled on Fluent CFD to simulate the applied test conditions. Numerical analysis was performed to compare the results with those from the experimental characterization. A numerical approach offers the advantage that a lot of parameters, including thickness, dimensions and thermal parameters can be varied. The interface resistance can also be tuned for a wide range of materials and applications. This chapter shows a detail description of geometrical design, modeling, simulation and analysis.

In order to conduct the numerical study for heat dissipation between a heat source and heat sink, two aluminum blocks were observed in contact in three ways;

1) **Dry Contact** – To understand the ideal contact condition with zero resistance.

2) **Using Arctic Silver as a TIM** – To validate the test model with experimental and published results.

3) **Using CNF buckypaper TIM** – To understand the effect of CNF on the global thermal resistance of the system.
In the model shown in Figure 3.35, the path of heat removal involves conduction across the interface of the upper aluminum block, through a TIM, into the lower aluminum block and then convection to the environment. For the system temperatures close to the ambient, natural convection and radiation terms were neglected.

The numerical analysis is based on the exact simulation of the test stand. The simulation is carried out based on the governing equations for the overall system. The heat flow is calculated as:

\[ Q = -A K \frac{dT}{dx} \]  

(1)

Figure 3.35 Schematic of the test model
Where;

K is the thermal conductivity and 
A is the surface area 

As shown in Fig 3b, the interface resistance is composed of three resistances in series. The total interface resistance is expressed as;

\[ R_{total} = R_1 + R_{cond} + R_2 \quad (2) \]

Where;

R\(_1\) is the contact resistance between TIM and the lower block, 
R\(_{cond}\) is the conduction resistance across the thickness of the TIM, 
R\(_2\) is the contact resistance between TIM and the upper block, and

\[ R_{cond} = \frac{t}{KA} \quad (3) \]

Where;

\( t \) is the thickness of the TIM, 

The total R can be expressed as \( R = R_1 + R_2 \). One thing to notice is that resistances \( R_1 \) and \( R_2 \) are most likely to be same since each of the two contacting surfaces are similar (aluminum). Hence the equation reads,
In order to carry out the model simulation in Fluent for the solid surfaces, the energy equation is given by:

\[ R_{\text{total}} = R + \frac{t}{KA} \quad (4) \]

\[ \frac{\partial}{\partial t} (\rho h) + \nabla \cdot (\vec{v} \rho h) = \nabla \cdot (K \nabla T) + H \quad (5) \]

Where:

\( \rho \) is the density,
\( h \) is the enthalpy,
\( K \) is thermal conductivity,
\( T \) is temperature and
\( H \) is the volumetric heat source.

For the heating plate, the input heat flux is given by:

\[ T_w = \frac{(q - q_{rad}) \Delta n}{K_s} + T_s \quad (6) \]

And the temperature boundary condition for the cooling plate is given by:

\[ q = \frac{K_s}{\Delta n} (T_w - T_s) + q_{rad} \quad (7) \]
Where;

\[ K_s = \text{Thermal conductivity of the solid}, \]
\[ T_s = \text{Local solid temperature}, \]
\[ \Delta \tau = \text{distance between wall surface and the solid cell center}. \]

3.5 Validation of the model

As a primary step, the numerical model was validated by comparing the results of the present numerical method using thermal paste TIM (Arctic Silver 5) with experimental results available in the literature. The values of total thermal resistance using this model showed practical agreement with the experimental and published results with an error of less than 6%. Table 1 compares the contact thermal resistance of Arctic Silver 5 at a given load and thickness.
Figure 3.36 Validation of the test model

Figure 3.36 shows the heat transport from the heating plate (50 W/m²) through the TIM (AS-5) and to the cooling plate (288 K). Four temperature reading at locations (T1, T2, T3 and T4) were obtained to calculate the total thermal impedance as a function of thickness of the TIM.
Table 3.12 Validation of the numerical model with experimental and published data for Arctic Silver 5

<table>
<thead>
<tr>
<th>Arctic Silver Thermal Interface Material</th>
<th>Load (kg)</th>
<th>Thickness (mm)</th>
<th>Thermal Impedance (K/W)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Published</td>
<td>135</td>
<td>0.0254</td>
<td>0.0136</td>
</tr>
<tr>
<td>Experimental</td>
<td>135</td>
<td>0.0254</td>
<td>0.0137</td>
</tr>
<tr>
<td>Numerical</td>
<td>-</td>
<td>0.0254</td>
<td>0.0133</td>
</tr>
</tbody>
</table>

Figure 3.37 Graph showing the thermal impedance comparison of Arctic Silver using various analysis techniques
Figure 3.37 shows the Arctic Silver 5 performance comparison of the values calculated from the numerical model with those obtained from the experimental setup and published results.

3.6 Numerical Simulation

Figure 3.38 shows the contours of static temperature change from the heat source to the heat sink. The efficient heat dissipation is simulated with the CNF buckypaper TIM. The heating plate was set to 60 W/m² and the cooling plate at 10 °C. The thickness of the TIM was 0.0254 millimeter. Two temperature
readings were obtained at contacting block to measure the thermal performance of the TIM.

Figure 3.39 shows the overall temperature distribution in terms of the position (geometry) of the test model. The close transition between the upper and lower block (green and red lines respectively), shows the reduction of air gaps resulting in the efficient heat transport path.

3.6.1 Parametric Study

For the designed model, the parametric study is carried out in terms of two parameters of the prepared thermal interface material.
a. Thickness of TIM

In an experimental setup, the thickness of the TIM decreases as the contact pressure increases. In order to duplicate this behavior in the numerical study, thickness of the TIM is taken into account as one of the influencing parameter in optimizing the total thermal performance. Five different thicknesses are simulated for each TIM. It is observed that as the thickness of the TIM decreases, more air gaps are filled and more heat is dissipated through the interface minimizing the total interfacial resistance. Figure 3.40 shows thermal impedance results obtained from the simulation on TIMs of different thicknesses.

![Figure 3.40 Thermal impedance of various TIMs as a function of thickness](image-url)
b. Thermal Performance of TIM

The simulations were performed to carry out a parametric study of CNF-TIMs in terms of thickness and thermal conductivity of the samples. Unlike experimental analysis, the applied load was duplicated as the thickness of the TIM. Hence, as the load increases, the thickness decreases and hence thermal impedance decreases (Figure 3.41). Overall results from the numerical analysis show a strong agreement with those obtained from the experimental analysis.
Chapter IV

Power Spike Analysis

Faster speeds for space vehicles, high power requirements for automotive engines, and brighter beams for optical devices, has resulted in excessive heating of the respective systems. In case of power electronics this quest for miniaturization has lead to the generation of very high heat fluxes (around $10^7 \text{W/m}^2$). The modern electronic and electrical systems are characterized by increase in the density of circuits within the chips. The denser the circuitry, the higher the power used to empower the chip and, consequently, the higher the heat generated. Intel-R microprocessors advanced from having 1.5 Am lines on INTEL386k [135] to 0.13 Am in the current PentiumR-4 [136] within a space of 7 years. This is more than ten-fold reduction in the line width. The increase in heat flow from IC chips is apparent in the trend of power dissipation for high-performance chips reported by the International Technology Roadmap for the Semiconductor (ITRS) [137–139]. Increasing amount of power dissipated has to be offset by efficient cooling because the maximum junction temperature stays at 85–90°C. A major challenge is the ability to manage the heat in the IC chips without compromising on the performance of the device. Thus, there is a growing
need to develop more effective TM techniques to control this high order of heat fluxes in different miniaturized devices. Conversely some of the electronic systems are subjected to transient heat loads. These pulsed heat inputs or sudden surge in heat flux called as ‘power spikes’, can cause significant temperature fluctuations. Thus the management of this thermal energy is very crucial because heat has many detrimental effects on the device. It has been reported [140] that failure rates have near exponential dependence on device temperature. Also the dominant failure in most power electronics systems is thermal related [141]. When a certain upper critical temperature is reached, important parts of the device may cease to function. Furthermore, temperature cycles that result from switching on and off the device can cause problems, even if the operating temperature does not hit the upper critical temperature. This leads to another problem, the reliability of the device, because temperature cycling of more than 20°C can increase the failure rate of a device by eight-fold [140]. There is therefore an additional challenge to the thermal manager: a need not only to dissipate heat from the device, but also to do it gradually. Thus, control of these power spikes or pulsed heat loads is one of the critical issues in the design of TM systems.

To meet the requirement on the safe operation area of the device, the thermal resistance of the device should be reduced by some kind of cooling. A number of researchers have used conventional interface materials to efficiently dissipate heat upon a sudden surge of power [142-147]. Baker et al. [142] performed a conduction-only analysis of the thermal performance of a
heterogeneous package. Two nonmetallic PCMs (octadecane and pentacosane) were used for heat storage with metal fins to enhance the effective thermal conductivity. Vesligaj and Amon [144] investigated passive thermal control using phase change materials during time-varying workloads on portable electronics. An epoxy polymer was used as a phase change material. They indicated that the operational performance of the portable electronics improved when such a passive thermal storage device was used. Binet and Lacroix [145] conducted a numerical study of natural convection-dominated melting inside a rectangular enclosure from three discrete heat sources. Evans et al. [146] analyzed power electronic packages and provided design guidelines relating the materials, geometry, power input and junction temperature for steady-state conditions and transient pulses. Krishnan and Garimella [147] carried out a fully transient analysis of the phase change process inside a rectangular enclosure for electronics cooling applications with pulsed power dissipation. The experimental investigation included the melting of a pure PCM, n-eicosane, inside a rectangular aluminum container with multiple discrete heat sources mounted on one side. A copper heat spreader was used in conjunction with the heat sources. The influence of changes in the frequency of pulses, heat source location and aspect ratio of the containment volume on the thermal performance of the PCM unit was studied. It was reported by the authors that the heat source location and container aspect ratio played a very significant role in the performance of the PCM system with pulsed heat input.
Carbon nanotubes (CNTs), because of their size and excellent thermal properties, have proven to be one of the most efficient materials for heat transport [148-152]. Xu and Fisher [148] tested thermal resistance of one sided CNT array interface (Si-CNT-Cu) under a pressure of 0.414 MPa with a pressure bar method and reported a thermal resistance of 20 mm$^2$ K/W. Thermal resistances between the free CNT array tips and an experimental contact were 17 and 15 mm$^2$ K/W at pressures of 0.040 and 0.100 MPa, respectively. Xu and Fisher [149] also combined thin layers of phase change material (PCM) with CNT arrays, and the composite produced a resistance of 5 mm$^2$ K/W under moderate pressures for Si–Cu interfaces. Wang et al. [150] used a photothermal technique to measure the thermal resistance between a CNT array and its growth substrate (Si-CNT). The resistance was relatively large, 16 mm$^2$ K/W, as the CNT array was of poor structural quality and no pressure was applied to the interface. Using a transient, Tong et al. [151] used a transient thermoreflectance technique to measure thermal resistance of one-sided CNT interface (Si-CNT-glass) and measured 18 mm$^2$ K/W. The authors concluded that the interface between the free CNT array tips and their opposing glass substrate (CNT-glass) dominated the total thermal interface resistance and suggested that this resistance could be further decreased by the application of pressure to the interface. Xu and Fisher [152] fabricated and experimentally studied two-sided CNT interfaces with CNT arrays directly synthesized on Si wafers and Cu blocks. With vertically aligned CNT arrays, an interface resistance less than 5 mm$^2$ K/W for two-sided CNT interfaces was measured. However, due to CNT growth and fabrication
constraints e.g., high-temperature substrate requirement, difficulties in fabricating samples with identical arrays on sides of a test chip [152], and high costs, there is an increased fabrication and measurement uncertainty, which sometimes can be larger than the magnitude of thermal performance.

Carbon nanofibers (CNF), though not that perfect in structure and relatively less conductive in nature, are fairly economical and do not require high temperature substrate for growth. These materials have been increasingly used for aircraft structures and need to be engineered with efficient lightning strike protection to achieve tolerance comparable to metallic components. Gao et al. [153] developed a paper made of carbon nanofibers and nickel nanostrands as a surface layer on the composite panels to explore their efficiency in transient power spikes. The porous, flexible, non-woven papers of nanofibers and nanostrands were prepared and incorporated onto the surface of carbon fiber reinforced polymer composites through resin transfer molding. Authors reported that the power strike tolerance correlated to the surface conductivities of composite materials and the surfaces of the materials were damaged due to the existence of organic binder. Ngo et al. [154] used electrodeposited Cu as gap filler to enhance the stability and thermal conductance of CNF arrays with a one-dimensional steady state reference bar method. The authors reported a thermal resistance of 25 mm$^2$ K/W under a pressure of 0.414 MPa for Si-Cu interfaces. These results show that CNFs have competitive thermal properties to those of CNTs with superiority in terms of cost and fabrication constraints. This section
explores the potential of self standing CNF buckypapers to sustain transient power spikes by efficiently dissipating maximum heat to the sink.

4.1 Experimental Setup

In order to test the thermal properties of various carbon-based TIMs, it was necessary to design a test setup. Many previous experimental set ups have been created to measure the thermal properties of TIMs [106-108]. However many of these set ups are expensive and time consuming. A test set up that was accurate, cost effective, and capable of rapidly generating usable data was created for this experiment. A similar setup was reconstructed and reprogrammed to meet the specifications required for the power spike analysis. Figure 4.1 shows the visual representation of the original test rig. The original design consisted of two main subassemblies; top plate with actuator and heater subassembly, coolant plate with sample holder and guide subassembly. The two subassemblies were connected to each other through two threaded aluminum rods. The upper heating plate consisted primarily of a strip heater with dimensions of 75 mm wide x 100 mm length attached to a Plexiglas plate and an aluminum plate with dimensions of 100 mm x 40 mm x 25 mm. This was connected to a 12 kg force actuator and a 120 watt heater. The lower assembly consisted of a cooling plate designed to maintain a constant ambient temperature. A “U” shaped copper pipe was inserted under the cooling plate which was attached to plastic tubing at each end for cold water supply.
The sample holder (figure 4.2) was designed with two aluminum blocks (heating and cooling plates) as contacting surfaces. Three holes were drilled down the center of each block. These thermocouple holes were drilled 10 mm deep. It was necessary to mount the blocks to the test rig. Two holes were made in each block, on the faces directly to the left and right of the thermocouples. These holes were drilled 8 mm deep in order to insure no interference with the thermal couples. The upper block was attached directly to the corresponding hot plate. For the bottom block, two more brackets were created and screwed into the aluminum block. These brackets were then attached to a support block located at the back of the test rig. The two blocks were aligned to insure an accurate contact area possible. To further insure optimal thermal conduction through the blocks, a high thermal conductivity paste (Omegatherm 201) was
spread at the interface of the surfaces of the blocks and their corresponding plates.

Figure 4.2 Schematic of the sample holder
The test setup was designed for thermal analysis of carbon nanofiber based thermal interface materials. The compliant buckypaper was placed on the lower block in the sample holder. The electromechanical actuator was used to apply and remove the strip heater on the top of the upper block as required. Thus, a thermal analysis of different samples could be performed by applying heat load at the top of block and dissipating the heat absorbed within the sample by a coolant flow at its bottom through the coolant plate. The above procedure was used for different techniques analyses such as measuring the temperature distribution within samples to analyze their heat transfer ability, TIM’s ability to handle maximum temperature, and the global thermal resistance. Figure 4.3 shows the experimental setup with the sample holder.
Before stepping into the actual analysis, the temperatures sensors were tested under dry test at ambient temperature. Six thermocouples were connected to the DAQ system and then inserted in two aluminum blocks (three in each) to verify the temperature. The thermocouple readings were noted for 10 minutes exposed to ambient conditions. Figure 4.4 shows the calibration analysis of six thermocouples as a function of time.

Figure 4.3 a) Experimental setup b) Enlarged view of the sample holder
The following equations are used to calculate thermal conductivity of a TIM assuming a one-dimensional heat flow through the thickness of the sample. The governing equation is obtained by using Fourier’s Law:

\[ k = \frac{q \cdot t}{A \cdot \Delta T} \quad \text{(Eq)} \]

Where:

- \( k \) = thermal conductivity
- \( q \) = heat flow (or power)
- \( t \) = interface thickness
\[ A = \text{area} \]
\[ \Delta T = \text{temperature difference across the interface} \]

However, it is important to know that the thermal conductivity of a TIM is solely a material property. In other words, the thermal conductivity reflects the type of material used; it does not account for any thermal effects caused by the interfacial resistance or the geometry of the sample. Hence, it is important to take into account the thermal effects on the interface of TIM and contacting surfaces. The global resistance can be determined by the below equation

\[
R = \frac{T_1 - T_2}{Q} = \frac{\Delta T}{Q}
\]

Where:

- \( R \) = thermal resistance
- \( T_1 \) = component temperature
- \( T_2 \) = heat sink temperature
- \( Q \) = heat flow (or power)

By using the equation of the thermal resistance, we can calculate each TIM's ability to resist sudden change in temperature. Therefore the significance of heat treatment on carbon structures can be highlighted by analyzing the change in temperature from heat source to the heat sink.
4.2 Results

Two tests were set up to examine two different types of heat transfer through each buckypaper TIM; a) the change in temperature between upper and lower surface which determines the ability of a TIM to effectively transport heat and b) maximum temperature reached at the upper surface which determines the ability of a TIM to handle high temperatures. To maximize the accuracy of the results, two thermocouple (closest to the interface) readings were examined. These tests were designed to imitate typical power demands put upon modern electronics.

4.2.1 Uniform Power Pulse - 24 watts of power was applied at the upper block for 60 minutes period. To obtain this, maximum power (120 watts) of power was applied for 5 seconds followed by a 20 second period with zero watts being applied. This gives 20% of the total power that is 24 watts. The heater was then turned off, applying zero power for an additional 25 seconds. This test simulates the power usage of a device that uses a constant power for a short period of time and then switch to stand by condition (does not utilize any power). This is common for many devices that are frequently turned on and off.

4.2.2 High Power Spikes - 12 watts of power was applied at the upper block for 60 minutes period. To obtain this, 120 watts of power was applied for 5 seconds followed by a period of 45 seconds with zero watts being applied. This gives 10% of the 120 watt heater that is 12 watts. This procedure was followed by a high power heat spike of 96 watts. To obtain that, 120 watts
of power was applied for 40 seconds followed by a period of 10 seconds with zero watts being applied. This gives 80% of the 120 watt heater that is 96 watts. These high power heat spikes simulate various demands put upon electronics. With most devices today, power fluctuates constantly and can experience instances of sudden surge of power.

In order to understand the effects of carbon nanofiber TIM compared the dry contact, and heat treatment effects of these TIM, both tests were run with;

**A) Direct contact (no TIM)**

Due to the rough nature of contacting surfaces (aluminum blocks in this case), the dry contact has an immediate disadvantage in terms of interfacial thermal resistance. Thermal resistance in dry contact therefore varies with respect to the evenness of contact surfaces which can never be perfect. Hence, in any case it can be very misleading and inaccurate to assume that the dry contact performs better than a TIM in real world applications. Figures 4.5 and 4.6 show the direct contact plots for uniform pulse and power spikes respectively.
Figure 4.5 Direct contact analyses for uniform heat pulse

Figure 4.6 Direct contact analyses for transient power spikes
In order to compare the results of the direct contact analysis, compliant TIMs of various heat treatment temperatures are used. Although the insertion of a compliant TIM adds a resistance to the system, but high conformability of the TIM leads to the enhancement of the global resistance of the system.

**B) Buckypaper TIM (CNF-HTT)**

![Buckypapers (CNF-HHT) - 24 Watt Uniform Pulse](image)

Figure 4.7 CNF-HHT buckypaper analyses for uniform heat pulse
Figure 4.8 CNF-HHT buckypaper analyses for transient power spikes

Three buckypaper TIMs composed of high heat treated (HHT) carbon nanofibers were tested at uniform heat pulse (figure 4.7) and transient power spikes (figure 4.8). The heat dissipation rate was observed in terms of temperature difference from the source to the sink. The maximum temperature values obtained for each TIM show the sustainability of a TIM at uniform and transient temperatures.
C) Buckypaper (CNF-LHT)

Figure 4.9 CNF-LHT buckypaper analyses for uniform heat pulse

Figure 4.10 CNF-LHT buckypaper analyses for transient power spikes
Two buckypaper TIMs composed of low heat treated (LHT) carbon nanofibers were tested at uniform heat pulse (figure 4.9) and transient power spikes (figure 4.10). The heat dissipation rate was observed in terms of temperature difference from the source to the sink. The maximum temperature values obtained for each TIM show the sustainability of a TIM at uniform and transient temperatures.

**D) Buckypaper (CNF-PS)**

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**Buckypapers (CNF-PS) - 24 Watt Uniform Pulse**

![Graph](image)

Figure 4.11 CNF-PS buckypaper analyses for uniform heat pulse
Two buckypaper TIMs composed of pyrolitically stripped (PS) carbon nanofibers were tested at uniform heat pulse (figure 4.11) and transient power spikes (figure 4.12). The heat dissipation rate was observed in terms of temperature difference from the source to the sink. The maximum temperature values obtained for each TIM show the sustainability of a TIM at uniform and transient temperatures.

Figure 4.12 CNF-PS buckypaper analyses for transient power spikes
Figure 4.13 Temperature comparison of various heat treated CNF buckypapers and direct contact subjected to uniform heat pulse.

Figure 4.14 Temperature comparison of various heat treated CNF buckypapers and direct contact subjected to transient power spikes.
Figure 4.13 and 4.14 show the overall temperature comparison of direct contact and CNF based TIMs for uniform heat and power spikes respectively. Two thermocouple readings were taken for each test to calculate the temperature difference.

Table 4.1 Maximum temperature results for uniform pulse analyses

<table>
<thead>
<tr>
<th>24 Watt Uniform Pulse over 60 min. period</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>TIM</strong></td>
</tr>
<tr>
<td>Dry Contact</td>
</tr>
<tr>
<td>CNF-HHT</td>
</tr>
<tr>
<td>CNF-LHT</td>
</tr>
<tr>
<td>CNF-PS</td>
</tr>
</tbody>
</table>

Table 4.2 Temperature difference results for uniform pulse analyses

<table>
<thead>
<tr>
<th>24 Watt Uniform Pulse over 60 min. period</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>TIM</strong></td>
</tr>
<tr>
<td>Dry Contact</td>
</tr>
<tr>
<td>CNF-HHT</td>
</tr>
<tr>
<td>CNF-LHT</td>
</tr>
<tr>
<td>CNF-PS</td>
</tr>
</tbody>
</table>
Table 4.3 Maximum temperature results for power spikes analyses

<table>
<thead>
<tr>
<th>TIM</th>
<th>Maximum Temperature (°C)</th>
<th>∆T (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dry Contact</td>
<td>132.82</td>
<td>Baseline</td>
</tr>
<tr>
<td>CNF-HHT</td>
<td>109.12</td>
<td>23.70</td>
</tr>
<tr>
<td>CNF-LHT</td>
<td>151.21</td>
<td>-18.39</td>
</tr>
<tr>
<td>CNF-PS</td>
<td>153.47</td>
<td>-20.65</td>
</tr>
</tbody>
</table>

Table 4.4 Temperature difference results for power spikes analyses

<table>
<thead>
<tr>
<th>TIM</th>
<th>Temperature Difference(°C)</th>
<th>∆T (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dry Contact</td>
<td>43.44</td>
<td>Baseline</td>
</tr>
<tr>
<td>CNF-HHT</td>
<td>19.95</td>
<td>23.49</td>
</tr>
<tr>
<td>CNF-LHT</td>
<td>80.08</td>
<td>-36.64</td>
</tr>
<tr>
<td>CNF-PS</td>
<td>86.11</td>
<td>-42.67</td>
</tr>
</tbody>
</table>

The most striking and notable inference made from the tables 4.1-4.4 is that buckypaper with high heat treated (HHT) nanofibers reduced the thermal resistance and enhanced heat transport in comparison to the dry contact. For the uniform heat pulse test, the maximum temperature of CNF-HHT-TIM is 46 Celsius and temperature difference between two surfaces was seen to be less than 5 Celsius. This shows that high heat treatment of carbon nanofibers allows the fibers to align in a columnar way and minimizes spacing between fibers. This allows an efficient path for heat to transfer from the source to the sink. For the
power spike test, the maximum temperature of CNF-HHT TIM is far better than that of the dry contact. Similarly, significant reduction in temperature difference states the ability to HHT-CNFs to resist sudden surge of power and efficient dissipation to the heat sink.

Thermal interface materials with low heat treated (LHT) and pyrolitically stripped (PS) fibers, however, show increase in thermal resistance and hence do not provide efficient heat path. Significant increase in thermal resistance is seen in these samples due to the randomness of the grown carbon nanofibers.

4.3 Summary

An experimental setup was designed and constructed to perform thermal analysis of carbon nanofiber based thermal interface materials. The setup consisted of four basic components; Main assembly, sample holder, data acquisition device and a control unit. The main assembly includes a heater plate with actuator, a coolant plate with sample holder. The sample holder consisted of two aluminum blocks as contacting surfaces attached to each side of the main assembly. The sample (TIM) was sandwiched between the two contacting blocks under test. A high performance analog/digital data acquisition system, Keithley 2700, was used to measure the power output from strip heater along with the required output data. Efficient data-logging software, ExceLINX, was used as an interface program for transferring the measured data from the DAQ. The pre-installed software was used to directly stream data from the DAQ board into the Excel. Software was programmed to analyze the data as it was received in Excel.
with Excel’s graphics, charting, and mathematical capabilities. An electronic control unit with the capability to control the speed of actuator along with its dwell time at the initial and retracted positions was used. The control circuit was programmed on MATLAB. Two detailed numerical codes were written for uniform heat pulse condition and spike power condition. The complete setup was calibrated and validated before actual tests.

Various heat treated carbon nanofiber based materials discussed in chapter 3 were used for detailed thermal analysis. All TIM samples were subjected to two types of heat loads; uniform pulse and transient power spikes. Uniform heat load of 24 Watts was applied over a period of 60 minutes. Transient power spikes of 96 Watts were applied over a period of 60 minutes. Results from all samples were compared to those of direct contact (without a TIM on the interface).

The above analysis emphasized the importance of using high heat treated carbon nanofiber based thermal interface materials. CNF-HTT sample outperformed all others with major enhancement in thermal transport from heat source to heat sink. This was due to the graphitization of fibers and its aligned nature at extremely high temperatures.
Chapter V
Conclusions

The present research is focused on the thermal management issues in electronics. These issues are typically caused by thermal interface resistance and pulsed heat loads. A comprehensive literature review provided in chapter 2, highlights these issues with existing solutions. Those solutions however have number of shortcomings that limit the overall performance of a system. By keeping those in view, a step wise process of carbon nanofiber based compliant thermal interface material (TIM) fabrication, experimental and numerical characterization and, analysis of real world applications is carried out. These processed materials are expected to overcome existing limitations by escalating the efficiency of the heat transport.

A detailed analysis was performed in chapter 3 to find the best material that could conform to the mating surface and minimize the thermal interfacial resistance. Various kinds of heat treated Carbon nanofibers (CNFs) were used namely 1) Pyrolytically stripped (PS), Light heat treated (LHT) and High heat treated (HHT). These nanofibers were mixed with a water soluble binder Polyvinyl Alcohol (PVA). The performance of CNFs was related to their degree of graphitizability. The higher the degree of graphitization and the better the heat transport across it. Various types of thermal interface materials were fabricated to
understand an efficient process to achieve pure carbon nanofiber materials with high thermal properties. The thermal properties of the final sample depend on the ratio of CNF to PVA, ultrasonic process time and number of agglomerates present in the solution.

The next step in the research was to design and construct an experimental setup that provides the characterization of the processed materials. The complete setup was validated using commercially available TIM (Arctic Silver 5) subjected to various thicknesses and loads. Actual experiments were performed without a TIM first (dry contact), and with fabricated TIM samples. The extension in the displacement cell measures the thickness of the TIM under test which is used to calculate the effective thermal conductivity of the TIM.

The main purpose of the test setup was to characterize the TIMs in terms of effects of the ratio of CNF and PVA on the global thermal resistance, and thermal impedance as a function of load and thickness. Four buckypaper samples with different thicknesses were tested in order to find the influence of those on global thermal resistance. It was found that as the load increases, TIM thermal conductivity increases with the load. This is due to significant decrease in TIM density. The heat transfer coefficient increases due to better conformability of the TIM at higher loads. The initial porosity of the buckypaper was about 40% but at higher loads, because of compression, carbon fibers tend to get closer allowing better heat transport. It is well known that the thermal conductivity of air is lower than that of carbon nanofibers and contact points increase with the
pressure, the thermal conductance through the thermal interface material increases.

For the thermal impedance comparison, the TIMs of approximately 0.1 mm thickness were selected. It was seen that the thermal impedance values with high heat treated CNF (HHT-CNFM) buckypaper were significantly better than low heat treated (LHT) and PS (pyrolytically stripped). For the buckypaper with HHT-CNFM as compared to the direct contact between two surfaces at maximum load, the thermal impedance was reduced by 54% from 0.151 K/W to 0.069 K/W.

In order to analyze the CNF to PVA weight ratio, four samples of CNF-HTT buckypaper were prepared. By varying the PVA weight and keeping the CNF weight constant (0.5g), different thermal impedance behaviors were seen. For each composition, two samples with different thicknesses were analyzed to find the thermal impedance as a function of thickness. It was noticed that two samples with same PVA to CNF ratio but different global weights show identical thermal behavior. It was hence important to define the samples by its PVA to CNF ratio and not its global weight. Hence the thermal impedance graph in terms of PVA to CNF ratio was analyzed which showed that at low pressures, as the ratio between two materials increased, the thermal impedance increased. However, the thermal impedance remains constant at high temperatures, regardless of the ratio between two materials.

The test setup was modeled using Fluent software package. The simulations were performed to carry out a parametric study of CNF-TIMs in terms of thickness and thermal conductivity of the samples. Overall results from the
numerical analysis show a strong agreement with those obtained from the experimental analysis.

The fabricated samples were then tested for real world power application using designed experimental setup. From the analysis, it was found that buckypaper with high heat treated (HHT) nanofibers reduced the thermal resistance and significantly enhanced heat transport in comparison to the dry contact. For the uniform heat pulse test, the maximum temperature of CNF-HHT-TIM was 47 Celsius and temperature difference between two surfaces was seen to be less than 5 Celsius. This shows that high heat treatment of carbon nanofibers allows the fibers to align in a columnar way and minimizes spacing between fibers. This allows an efficient path for heat to transfer from the source to the sink. For the power spike test, the maximum temperature of CNF-HHT TIM was far better than that of dry contact, taking high contact points into account. Similarly, major reduction in temperature difference states the ability to HHT-CNFs to resist sudden surge of power and efficient dissipation to the heat sink.

Thermal interface materials with low heat treated (LHT) and pyrolytically stripped (PS) fibers, however, show increase in thermal resistance and hence do not provide efficient heat path. Significant increase in thermal resistance is seen in these samples due to the randomness of the grown carbon nanofibers.

The above analysis emphasized the importance of using high heat treated carbon nanofiber based thermal interface materials. CNF-HTT sample outperformed all others with more than 50% enhancement in thermal transport
from heat source to heat sink. Because of their graphitic nature, these are highly conductive and hence can be used as a potential TIM for aerospace applications.

The overall research showed that in comparison to existing thermal interface materials like thermal pastes and greases, carbon nanofiber based materials are dry in nature and compliant enough to conform to the mating surfaces. Because of high thermal properties of carbon nanofibers, these materials provide better thermal transport from heat source to heat sink. The added advantages of these materials are these are installation friendly and can perform at extremely high temperatures without breaking or leaking out of the interface.
Chapter VI

Recommendations

Carbon Nanotubes (CNTs) have received significant attention among the researchers [87-98] because of its small diameter and ultra high thermal properties. The properties of these nanostructures can further be enhanced by altering its size and orientation of the tubes. As a next step to this research, CNFs can be replaced by aligned CNTs to maximize the heat dissipation across the interface. CNTs can be grown on a substrate than can sustain temperatures of 700 °C or more, and peeled off to be used as a self standing CNT buckypaper. The aligned CNTs generate a fairly good adhesive force which can act as an efficient heat transport from source to sink. Figure 6.1 shows the forces required for a gecko to climb up the vertical wall. The toes of the gecko have a special adaptation that allows them to adhere to most surfaces without the use of liquids or surface tension. The spatula tipped setae on gecko footpads demonstrate that the attractive forces that hold geckos to surfaces are Van der Waals interactions between the finely divided setae and the surfaces themselves [155]. These Van der Waals interactions are strong enough to support up to 100 times the animals’ bodyweight. Liehui [156] fabricated macroscopic patches of compliant vertically aligned CNT array. The author explained that the fabricated patches of uniform
array had adhesive strength similar to that of geckos (10 N/cm²) on a variety of substrates and could be easily removed by peeling. When the array was patterned to mimic the hierarchical structures of gecko foot-hairs, strength was increased up to four times. The author reported that those patches were self-cleaning, non-visco-elastic and gave good strength in vacuum. Since these properties are highly desired in thermal management, microelectronics and space operations, CNT array can be a good candidate for efficient heat dissipation.

Figure 6.1 Adhesive forces of a single gecko foot [157]
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APPENDIX

Figure A-1 Cooling Plate

Figure A-2 Aluminum Base Plate
Figure A-3 Heater Plate

Figure A-4 Plastic Insulator
Figure A-5 1x1 inch Aluminum Block

Figure A-6 2x2 inch Aluminum Block
Figure A-7 Test Setup - 1x1 inch blocks installed

Figure A-8 Test Setup - 2x2 inch blocks installed