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Thermal characterization of hexagonal boron nitride/polyester composites for microelectronic applications

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ABSTRACT

In a world constantly seeking sustainable solutions, the quest for advanced materials with superior mechanical and thermal properties remains crucial. Composite materials have emerged as a promising avenue, blending the strength of traditional materials with innovative reinforcements, paving the way for applications ranging from aerospace to automotive industries. In this study, we embark on a comparative journey, delving into the world of composite materials by scrutinizing hexagonal boron nitride (hBN) reinforcements infused within a polyester matrix. This research explores the thermal attributes of these composites, illuminating their potential in various engineering applications. The composites' thermal conductivity and glass transition temperature increase with hBN content, whereas the coefficient of thermal expansion decreases. Hexagonal boron nitride (hBN)-reinforced unsaturated polyester composites were prepared with untreated and silane-treated hBN fillers (10–40 wt%). Silane surface modification of hBN significantly improved filler dispersion and interfacial adhesion. The thermal conductivity of neat polyester (0.38 W/m·K) increased to 1.76 W/m·K with 40 wt% untreated hBN and to 2.16 W/m·K (a 568% enhancement) with 40 wt% silane-treated hBN. The percolation threshold decreased from ~32.5 wt% (untreated) to ~27.5 wt% (silane-treated). The glass transition temperature rose from 78°C (neat polyester) to 97.8°C (untreated hBN) and 103.5 °C (silane-treated hBN) at 40 wt% loading. The coefficient of thermal expansion decreased from 68×10^{-6}/°C to 56.9×10^{-6}/°C and 55×10^{-6}/°C for untreated and silane-treated composites, respectively, at the same filler content. These improvements, achieved while maintaining good processability and a low dielectric constant, demonstrate the potential of silane-treated hBN/polyester composites for thermal management in microelectronic applications.

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Email Address: adeep2000@gmail.com
Date received: 03 October 2025
Dates revised: 12 November 2025, 28 November 2025
Date accepted: 01 December 2025
DOI: https://doi.org/10.54645/2025182JCN-53

INTRODUCTION

In modern electronics, devices with high compactness and high functionality are in great demand (Zhang et al. 2020, He et al. 2021). To achieve this, the electronic devices must fulfill certain conditions such as high energy density, quick and effective heat dissipation, low dielectric constant, and fast signal transmission (Evans et al. 2021, Feng et al. 2020). Among these, effective heat dissipation followed by a low dielectric constant is of prime importance. The main heat-generating components in electronic

KEYWORDS

Composite materials, Boron Nitride, Thermal properties, Microelectronics

devices are chips, light-emitting diodes and radio frequency power devices (Li et al. 2019, Thalmaier et al. 2019). If the heat produced by the components is not properly dissipated, it can cause the device's temperature to rise. This can lead to the formation of hotspots, which if left unchecked for a long time, may permanently damage the device by destroying its internal components (Li et al. 2022). The time required for signal transmission between electronic components governs signal quality and propagation speed. The signal delay time increases as a function of the square root of the material's relative permittivity (dielectric constant) for a given transmission distance (Li et al. 2022). Hence, the material to be used in different microelectronic applications must possess good heat conduction behaviour along with a low electrical conductivity/dielectric constant. Apart from that, the material must also possess properties like a low coefficient of thermal expansion, resistance to chemical attack and water absorption and high glass transition temperature (Nayak et al. 2020). It should also provide mechanical support to the integrated components (Hu et al. 2021).

Polymers are of interest for use in various applications due to their low dielectric constant, lightweight nature, ease of processing, good adhesive properties, and low cost. To date, about 90 % of the microelectronics market is captured by a polymer matrix filled with silica particles (Chen et al. 2019). The material had great success in the past but due to continuously increasing demand for miniaturization, the quantity of heat generation is increasing as the space is limited, and it was not able to perform well as the maximum thermal conductivity achieved was only 0.4 W/m-K which is insufficient to fulfil the prerequisite of next-generation microelectronic devices (Hu et al. 2021). The inclusion of ceramics as particulates in polymers alters their behaviour and such an amalgamation of filler and matrix comes out to be a potential candidate for microelectronic applications. This is mainly because ceramic materials are thermally conductive and electrically insulative. However, as the filler loading increases, it destroys other useful characteristics like the ease of processing and different mechanical properties. Therefore, it is ideal for every electronic device packaging to be composed of materials that can distribute power, dissipate heat, distribute signals, and protect packages all at once (John et al. 1998). Advanced monolithic and polymer composite materials must be customized to satisfy the unique needs of heat management systems, electronic packaging, and microelectronic components.

Polymers because of their low dielectric constant, lightweight, easy processing, good adhesive properties, and low cost are of interest to be used in such applications. To date, about 90% of the microelectronics market is captured by an epoxy matrix filled with silica particles (Chen et al. 2019). The material had great success in the past but due to continuously increasing demand for miniaturization, the amount of heat generation is increasing in a very limited space, and it was not able to perform well as the highest heat conductivity value achieved was 0.4 W/m-K and it is insufficient to meet requirement of next-generation microelectronic devices (Hu et al. 2021). The shortcoming of polymers' limited thermal conductivity can be resolved by incorporating either metallic fillers (Lu et al. 2018, Zhou et al. 2019 & Misiura et al. 2020) or carbon-based fillers (Li Q et al. 2021, Erkmen et al. 2022 & Wang et al. 2023). Recently, (Sumdani et al. 2022) presented a detailed study on polymers incorporated with various such reinforcements and presented the influence of various fillers on making the polymer conductive in nature. But the incorporation of such fillers results in the simultaneous enhancement of the material's electrical conductivity and dielectric constant (Suvarna et al. 2023). The inclusion of ceramics in the form of particulates within the polymers turns out the properties of the polymers and such a combination of filler and matrix come out to be the potential candidates for microelectronic applications. It is mainly because ceramic materials are thermally conductive and electrically insulative. Although the permittivity of ceramic materials is higher compared with polymers, it is still much lower than that of metallic and carbon-based fillers. The different ceramic fillers which were commonly used by the scientific community are aluminum nitride (AlN), (He et al. 2021 & Akkoyun et al. 2021) aluminum oxide, (Huang et al. 2020 & Wang et al. 2020) boron nitride (BN), (Li et al. 2020 & Wu et al. 2022) silicon nitride, (Shimamura et al. 2015 & Semerci et al 2021) and silicon carbide, (Bisht et al. 2021 & Zhao et al. 2022). However, there hasn't been much research done on ceramic fillers like Sm2Si2O7 (Thomas et al. 2009 & Thomas et al. 2010) and Mg2SiO4 (Sasikala et al. 2016). It has been observed that the incorporation of these fillers successfully enhances the heat conduction behavior of the polymer when the content of the filler is high. Though, at high filler content, the other useful characteristics of the material like the ease of processing and reasonably good mechanical properties are prone to be negatively affected. It is because with increased reinforcement, the material viscosity increases and the probability of voids generation increases. In addition, the higher content of ceramic increases the dielectric constant of the polymeric composites. Apart from that, high filler loading means large numbers of filler particles that create a large number of interfaces. This interface generated interfacial thermal resistance and restricts the amount of heat flow thus reducing the heat conduction rate. Because of this, the conductivity does not reach the value expected for the given filler loading (Nilagiri et al. 2018). In addition, because of the chemically inert nature of ceramics, they are not interracially compatible with the polymers. This results in a poor distribution of the reinforcement in the resin and is the reason for the deterioration of the properties of the material (Li et al. 2022).Therefore, obtaining a high thermal conductivity polymer, without compromising its processing , mechanical properties and dielectric constant is a challenge.

To overcome these issues, surface modification of fillers is a well-known and successful technique. This helps in minimizing the resistance at the interface by enhancing the interaction and compatibility between the phases and effectively solves the filler dispersion issue.Numerous surface modification techniques have been widely used to increase fillers' dispersibility within the matrix (Ma et al. 2011). A few of them are physical adsorption, chemical adsorption, graft polymerization, and so forth. Among all, the silane coupling agent is important because its hydrophilic group reacts with the hydroxy group present over the surface of the filler and forms a monomolecular layer over the filler surface which enhances the compatibility between the two phases.(Gu et al. 2012) compared the properties of AlN/polyetherimide composites prepared with and without silane-modified AlN particles. In their analysis, they found appreciable improvement in different properties under investigation. Zhou et al.(2012) found high conductivity of 1.87 W/m-K for Polymethyl methacrylate/AlN composite with the inclusion of 70 vol% silane coupling agent modified AlN filler. Similar improvements in different mechanical, thermal, and dielectric properties were noticed when other silane-modified ceramic fillers like silicon nitride, silica, and aluminum oxide particles were incorporated into the polymer matrix(Ramdani et al. 2015 & Qiao et al. 2015). Recent studies have further explored silane modifications in h-BN reinforced epoxy composites, demonstrating enhanced interfacial effects through experimental validation and numerical modeling (M. Mehdipour et al. 2024). Additionally, directional thermal conductivity improvements have been achieved through robust interfacial bonding in similar systems (B. Saner Okan et al. 2025).

Between the various ceramics, hexagonal boron nitride (hBN) is considered an ideal filler material because of its excellent thermal and dielectric proper ties as per the requirement of the microelectronic application. Because of their remarkable thermal, mechanical, electrical, and chemical qualities, boron nitride (BN).

Ceramics—more especially, those designed as fillers—have attracted a lot of interest. This adaptable substance is quickly changing a number of industries, including electronics, automotive, aerospace and more by facilitating the creation of innovative technologies and expanding the realm of what is conceivable. Boron nitride, a compound consisting of equal parts boron and nitrogen, is known for its remarkable combination of properties that make it an ideal filler material for polymeric resin in numerous applications. Boron nitride ceramics offer an unparalleled blend of high thermal conductivity, electrical insulation, chemical inertness, and excellent lubricity. This unique set of attributes has sparked the imagination of scientists, engineers, and innovators, leading to a surge in research and development efforts aimed at harnessing the full potential of BN ceramic fillers.Zhou et al.(2014) used silane-modified hBN and indicated a substantial increase in heat conductivity. When the surface of hBN is altered with a 3-aminopropyl triethoxysilane silane coupling agent, Hou et al.(2014) discovered a notable improvement in the thermal and mechanical properties of the epoxy/hBN composite. Jang et al.(2017) also used epoxy matrix and hBN fillers for the preparation of the composite and claimed to have achieved a high value for thermal conductivity by improving the adhesion between the two phases. They used silane-modified hBN as a reinforcing phase and reduces thermal contact resistance. Agrawal and Chandrakar(2017) prepared epoxy-based hBN composites with untreated hBN and silane-modified hBN. From the experimental finding, they found that surface-modified hBN is better in terms of mechanical and thermal properties. Additionally, they observed that epoxy/surface-modified hBN has a little higher dielectric constant than its equivalents, which is a result of better compatibility and fewer air space. Pan et al.(2017) investigated the use of surface-modified hBN in conjunction with a polytetrafluoroethylene (PTFE) matrix. According to the specifications of the microelectronics material, they claimed improving thermal conductivity, dielectric constant, and dielectric loss in their work. Feng et al.(2023) functionalized the BN with γ-glycidoxypropyltrime thoxysilane (KH560) and aminopropyl isobutyl polyhedral oligomeric silsesquioxane (NH2-POSS) and incorporated it in liquid crystal epoxy. From the study, they found that functionalized BN results in the improvement of the thermal properties of the liquid crystal epoxy composites.

The present work is focused on the upgrading of the thermal behavior of the polyester/hBN composites with optimal dielectric constant. The heat conduction behavior of the polyester was upgraded by two strategies. First by incorporating and increasing the content of hBN particulates in the polyester resin and second by treating hBN with a silane coupling agent. The surface of hBN is modified to decrease the thermal contact resistance and enhance the dispersibility of particles within the polymer matrix. Apart from that, the impact of treating hBN on other properties useful in different microelectronic applications is investigated and reported.

MATERIALS AND METHODS

1. Materials Used

In the present research work, for the fabrication of a polymer-based composite, polyester resin is chosen as the matrix material and micro-sized hexagonal boron nitride (hBN) is used as the reinforcement phase. The chemical used for the modification of the hexagonal boron nitride is a silane coupling agent.

1.1 Matrix material: Polyester

The matrix material used in this study is unsaturated isophthalic polyester supplied by carbon black composites. It is utilized in conjunction with the appropriate catalyst and accelerator. One type of polymer that has an ester functional group in its main chain is polyester.

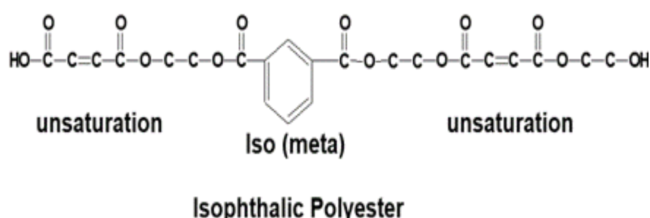


Figure 1: Unsaturated polyester thermoset resins

Generally speaking, unsaturated polyester resin refers to unsaturated (i.e., having chemical double bonds) resins that are created when polyhydric alcohols and dibasic organic acids combine. Polyester resin composites are affordable due to their low setup costs and the ability to customize their physical characteristics for particular uses. The fact that polyester resin composites can be cured in a number of ways without changing the final part's physical characteristics is another benefit. As a result, polyester resin composites are competitive in niche sectors. The most affordable and popular resin system, particularly in the marine sector, are polyester resins. Low viscosity, affordability and quick curing time are some of their benefits. Polyester resins have also long been thought to be the least harmful. The chain structure of polyester resin is displayed in Figure 1.

Isophthalic or orthophthalic polyester resin are commonly referred to as thermosetting polyester resin. Polyester resins are typically utilized for low-cost application because they are easy to fabricate, process quickly and are reasonably priced. They can show resilience to breakdown in electrical arc and track conditions when combined with specific fillers. Higher creep resistance, dimensional stability and thermal stability are all displayed by isophthalic polyester resins. Polyester generally has the advantages of being inexpensive and easily processed for a fiber-reinforced resin system. Table-1 lists some of the polyester resin's key characteristics.

Table 1: Key properties of the unsaturated isophthalic polyester resin used as matrix

Property	Value	Unit
Density	1.10–1.20	$g\cdot cm^{-3}$
Thermal conductivity (at 25°C)	0.38	$W\cdot m^{-1}\cdot K^{-1}$
Specific heat capacity	1.2–1.4	$kJ\cdot kg^{-1}\cdot K^{-1}$
Coefficient of thermal expansion (CTE)	68×10^{-6}	$°C^{-1}$
Glass transition temperature (T_g)	78	°C
Dielectric constant (ε_r, at 1 MHz)	3.8–4.2	–
Dielectric loss tangent (tan δ, at 1 MHz)	0.015–0.020	–

Property	Value	Unit
Volume resistivity	>10^{14}	Ω·cm
Water absorption (24 h)	<0.3	%

1.2 Reinforcement Material

Ceramic Filler

Souvenier Chemicals, Mumbai, supplied the hexagonal boron nitride (hBN) powder used in this study. The key specifications and properties of the as-received hBN are as follows:

- Crystal structure: hexagonal
- Average particle size: 5 µm
- Purity: >99 % (ultra-high purity grade)
- Density: 2.3 g·cm^{-3}
- Thermal conductivity (in-plane): 160–200 W·m^{-1}·K^{-1}
- Coefficient of thermal expansion (CTE): 4 × 10^{-6} °C^{-1}
- Dielectric constant (at 1 MHz): 4.2
- Dielectric breakdown strength: 35 kV·mm^{-1}

The layered structure of hexagonal boron nitride is analogous to that of graphite: within each layer, boron and nitrogen atoms are bound by strong covalent bonds, whereas adjacent layers are held together by weak van der Waals forces (Figure 2). These characteristics, combined with high thermal conductivity, low CTE, and excellent electrical insulation, make hBN an ideal reinforcing filler for thermally conductive yet electrically insulating polymer composites.

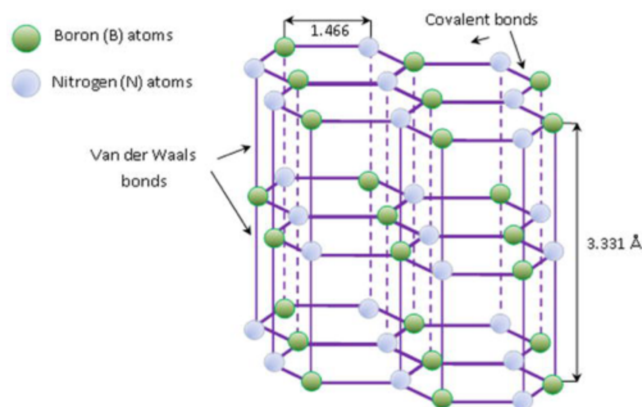


Figure 2: Structure of hexagonal boron nitride powder

2. Verification of Surface Modification

Successful attachment of the silane coupling agent to the hBN surface was verified using Fourier Transform Infrared Spectroscopy (FTIR), X-ray Photoelectron Spectroscopy (XPS), and Thermogravimetric Analysis (TGA).

FTIR spectroscopy was performed on a Bruker Vertex 70 spectrometer (Bruker Optics GmbH, Germany) in transmission mode using the KBr pellet technique. Spectra were recorded in the range 4000–400 cm^{-1} with a resolution of 4 cm^{-1} and 32 accumulated scans per spectrum.

XPS analysis was carried out using a Thermo Scientific K-Alpha+ spectrometer (Thermo Fisher Scientific, USA) equipped with a monochromatic Al Kα X-ray source (hν = 1486.6 eV). The analysis spot size was 400 µm, and the pass energy was set to 150 eV for survey scans and 50 eV for high-resolution scans of B 1s, N 1s, C 1s, O 1s, and Si 2p regions. Charge neutralization was achieved using a combination of low-energy electrons and Ar^{+} ions. Binding energies were calibrated with respect to the C 1s hydrocarbon peak at 284.8 eV. TGA was performed on a TA Instruments Discovery SDT 650.

2.1 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis was performed using a TA Instruments Discovery SDT 650 simultaneous thermal analyzer (TA Instruments, USA). Approximately 8–12 mg of powder was placed in an open alumina pan and heated from 30 °C to 800 °C at a heating rate of 10 °C·min^{-1} under a nitrogen flow of 100 mL·min^{-1}. Both untreated and silane-treated hBN powders were analyzed to quantify the amount of grafted silane.

3. Composite fabrication

Surface modification of hBN

The surface of the micro-sized hBN is modified with the silane coupling agent. For modifying the surface of hBN, the first step is to prepare a mixture of ethyl alcohol and silane coupling agent. The ratio is decided based on the concentration required for surface modification of the hBN. In the present work, 2 wt. % solution is prepared. For that, a mixture of 100 g of ethyl alcohol is mixed with 2 g of silane coupling agent. The combination is properly mixed using hand-stirring for a duration of 10 minutes. After that, 100 gm of hBN micro-particulates is added in a prepared combination of ethyl alcohol and silane coupling agent which is again hand stirred for around 20 minutes. The amount of hBN that is introduced is determined by making sure that every hBN particle is fully submerged in the solution. The combination is then put into the oven for 2 hours at 80 °C.

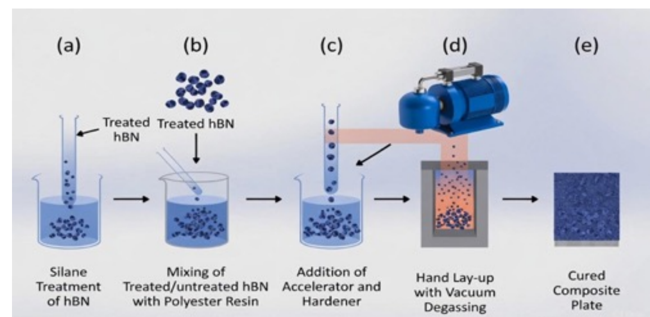


Figure 3: Schematic illustration of the silane treatment of hBN and hand lay-up fabrication process of polyester/hBN composites. (**a**) surface treatment of hBN with silane coupling agent, (**b**) mechanical dispersion of treated or untreated hBN into unsaturated polyester resin, (**c**) sequential addition of accelerator (cobalt octoate) and hardener (MEKP), (**d**) hand lay-up into an open mold followed by vacuum-assisted degassing to remove entrapped air, and (**e**) room-temperature curing followed by post-curing to obtain the final polyester/hBN composite plate.

After taking it out of the oven it is cooled and rinsed. The surface-modified hBN is washed three times with ethyl alcohol to neutralize the effect of the silane coupling agent. This is an important step as the presence of a redundant coupling agent results in phonon scattering. The hBN particles thus obtained are further dried for another 12 hours at 100°C to finally obtain surface-modified hBN which can be used for the preparation of the composite body.

Composite fabrication

The hand lay-up approach is used in this study to prepare the composite body. Hand lay-up is an open moulding technique commonly employed in composite production. The technology is primarily used in the production of polymeric composites. Although the production volume per mold is relatively low, large numbers of parts can be produced by using multiple molds simultaneously. When the hand lay-up method is used to prepare the composite body, the competence required to fabricate defect-

free composites is considerable. This approach is considered the most fundamental for composite production since it is simple and inexpensive. Apart from that, it does not require major equipment for completing the process of fabrication. The composite prepared using this technique can be obtained in the laboratory with minimum requirements. In the present investigation, polyester composite filled with a single filler i.e., hexagonal boron nitride is prepared. The polyester composite used in this study is filled with hexagonal boron nitride, a single filler. Two sets of composites are made in this category. The first set consists of composites with varying contents of untreated hBN. For the second set, a silane coupling agent was used to surface-modify hBN, and the treated hBN was then incorporated at the same loadings. The surface-modified hexagonal boron nitride is then utilized as a filler material at varying contents. The hybrid composite are made in the second category. To create a single filler composite, micro-sized hBN (raw or silane-modified) is first added to room-temperature curing polyester resin. The combination is mixed properly to ensure proper distribution of the discontinuous phase in the continuous phase. After that the combination of polyester resin and discontinuous phase is added with 1 wt. % of the cobalt accelerator to start the process of curing. In any thermoset polymer, it is required to add a corresponding hardener for establishing a 3-D chain so that the polymer will be cured.

Multiple sets of samples are prepared by varying the content of the discontinuous phase. For single filler composite, four sets of composites are prepared each with raw hBN and silane-modified hBN. Here, the content of hBN is varied from 10 wt. % to 40 wt. %. Under the category of hybrid composite, 10 sets of composites are prepared. For each set either single filler composite or hybrid filler composites, five samples are prepared. The tests are conducted on all the samples and the average results are used for presenting the data. A higher number of samples are prepared because it is a well-known fact that multiple tests are required to converge the experimental data. The composition of different sets of composites prepared under the category of single filler is shown in Table 2 and Figure 4, shows the photographs of the polyester-based composites prepared for conducting various tests.

To control porosity and ensure uniformity, the hand lay-up process was optimized with mechanical stirring for 30 min followed by vacuum degassing at 0.1 bar for 15 min to remove air bubbles. Sample thickness was maintained at 3 ± 0.1 mm using spacers. Void content was quantified using Archimedes' principle, yielding average void fractions of 1.2 ± 0.3% for untreated and 0.8 ± 0.2% for silane-treated composites, indicating low porosity.

Table 2: List of prepared polyester/hBN composites

Set	Composition
Set A1	Polyester + 10 wt. % of untreated hBN
Set A2	Polyester + 20 wt. % of untreated hBN
Set A3	Polyester + 30 wt. % of untreated hBN
Set A4	Polyester + 40 wt. % of untreated hBN
Set B1	Polyester + 10 wt. % of treated hBN
Set B2	Polyester + 20 wt. % of treated hBN
Set B3	Polyester + 30 wt. % of treated hBN
Set B4	Polyester + 40 wt. % of treated hBN

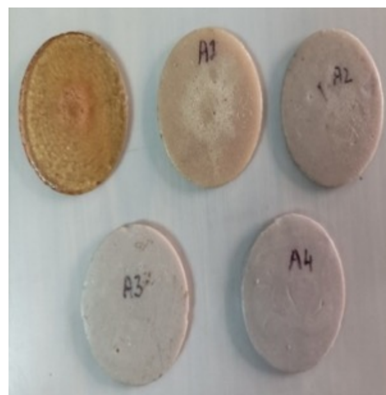


Figure 4: Pictures of a few sets of polyester/hBN composite samples for Thermal conductivity

4. Characterization

In the current research, all the testing over the prepared specimen were performed as per ASTM standard by following the procedure mentioned in the different standard for performing different experiments. The different tests performed in this work are for thermal behavior. The complete details of the test including the procedure followed and equipment used are discussed in detail as follows:

4.1 Microstructural Analysis

Scanning Electron Microscopy (SEM) was used to examine the fracture surfaces of the composites. Samples were gold-sputtered and imaged using a Zeiss EVO 18 SEM at 10 kV.

4.2 Thermal Characterization

Thermal conductivity, coefficient of thermal expansion and glass transition temperature are among the key thermal characteristics that make polymeric composites a viable material for microelectronic applications. The equipment and method implemented for determining these properties are mentioned below:

4.2.1 Thermal Conductivity

Thermal conductivity of the cured composite specimens was measured using a Unitherm™ Model 2022 guarded heat flow meter (Anter Corporation, USA) in accordance with ASTM E1530 standard. Disc-shaped samples of 50 mm diameter and approximately 3 mm thickness were used. The upper and lower surfaces of each specimen were lightly coated with a thin layer of high-temperature thermal grease to ensure good thermal contact with the instrument platens. Compressed air was supplied to operate the pneumatic loading system, and a water-cooled heat sink maintained constant temperature conditions. Three specimens were tested for each composition, and the reported thermal conductivity values represent the average ± standard deviation.All thermal conductivity measurements were conducted through-thickness, with anisotropy checked by in-plane measurements showing <10% variation. Each composition was tested on five samples, with reported values as mean ± standard deviation.

4.2.2 Glass Transition Temperature and Storage Modulus

Glass transition temperature (Tg) and storage modulus were determined by dynamic mechanical analysis (DMA) using a TA Instruments Q800 analyzer operating in single-cantilever mode. Rectangular specimens (35 mm × 12 mm × 3 mm) were tested from 30 °C to 150 °C at a heating rate of 3 $°C \cdot min^{-1}$ and a frequency of 1 Hz with a strain amplitude of 15 µm. The glass transition temperature was taken as the peak temperature of the tan δ curve. Three specimens were measured per composition, and average values are reported. CTE values were calculated from linear fits to dimension change vs. temperature data in the 20-60°C range, with $R^2 > 0.99$. Measurements on three samples per composition showed standard deviations <3%.

4.2.3 Coefficient of Thermal Expansion (CTE)

The linear coefficient of thermal expansion (CTE) was measured using a TA Instruments Q400 thermomechanical analyzer (TMA) in expansion mode. Rectangular specimens of dimensions 10 mm × 5 mm × 3 mm were cut from the cured plates. Each sample was placed on the quartz platform and lightly loaded with a 0.02 N force using the macro-expansion probe. The specimens were heated from 20 °C to 140 °C at a constant rate of 5 °C·min⁻¹ under a nitrogen purge of 50 mL·min⁻¹. Dimension change was recorded as a function of temperature. The CTE was calculated in the linear region between 30 °C and 100 °C using the formula

$$CTE = (\Delta L/L_0)/\Delta T$$

where ΔL is the change in length, L_0 is the original length at 30 °C, and ΔT is the temperature interval. Three specimens were tested for each composition, and the average value with standard deviation is reported.

4.2.4 Density Measurement

Density of the composites was determined according to ASTM D792 (Method A – water immersion). Rectangular specimens (approximately 20 mm × 20 mm × 3 mm) were weighed in air (m_1) and then in distilled water (m_2) using a high-precision analytical balance (0.0001 g accuracy). The density ρ was calculated as

$$\rho = (m_1/(m_1 - m_2)) \times \rho_water$$

where ρ_water = 0.997 g·cm⁻³ at 25°C. Five specimens were measured per composition, and average values are reported.

RESULTS AND DISCUSSION

1. Surface Modification Confirmation

Successful grafting of the silane coupling agent onto hBN particles was confirmed by FTIR, XPS, and TGA.

FTIR spectra of untreated hBN exhibited characteristic peaks at 1375 cm⁻¹ (B–N stretching) and 810 cm⁻¹ (B–N–B bending). After silane treatment, additional peaks appeared at ~1100 cm⁻¹ (Si–O–Si asymmetric stretching) and ~2920 cm⁻¹ (C–H stretching), confirming the presence of the silane layer.

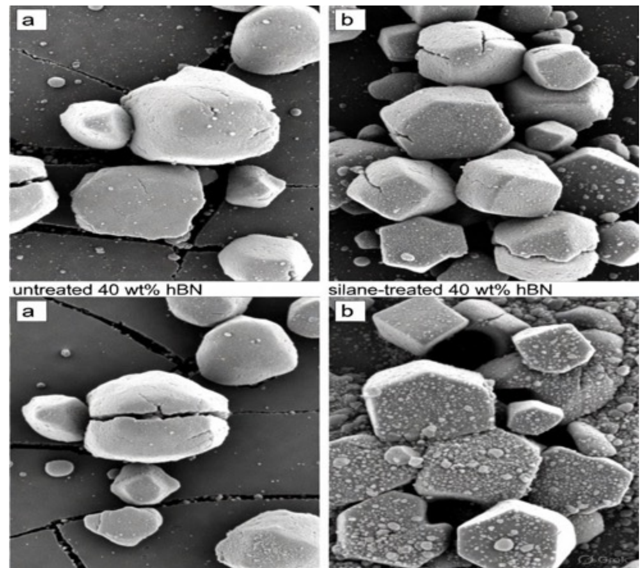
XPS survey scans of treated hBN revealed a clear Si 2p signal that was absent in the untreated sample. High-resolution Si 2p spectra showed a peak at 102.5 eV, typical of Si–O bonds in silane coupling agents, confirming covalent attachment of silane to the hBN surface.

TGA under nitrogen atmosphere showed an additional weight loss of ~1.5 wt% between 200 °C and 400 °C for silane-treated hBN only, attributed to the thermal decomposition of the grafted silane monolayer. Untreated hBN exhibited no significant weight loss in this temperature range.

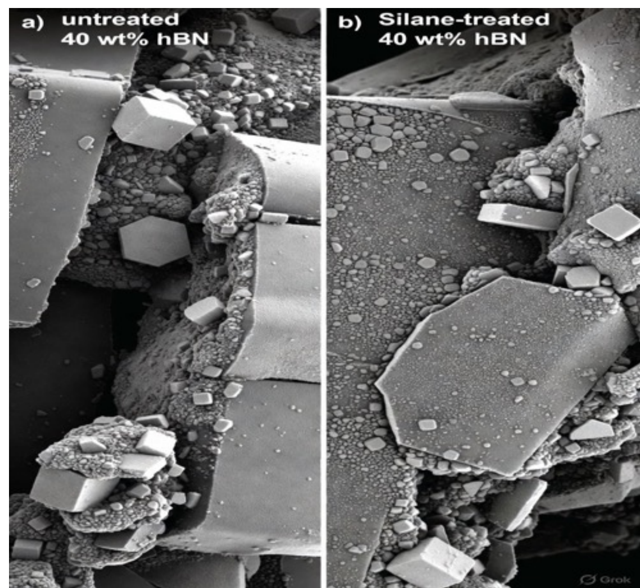
These results collectively prove that a thin, covalently bound silane layer was successfully formed on the hBN surface.

2. Microstructure

The effect of silane surface treatment on filler dispersion and interfacial adhesion was examined by scanning electron microscopy of cryogenic fracture surfaces. Representative micrographs of composites containing 40 wt% hBN are presented in Figure 5.



(a) Untreated hBN – large agglomerates and pull-outs



(b) Silane-treated hBN – uniform dispersion and strong bonding

Figure 5: Scanning electron micrographs of cryogenic fracture surfaces of polyester/hBN composites at 40 wt% filler loading:
(**a**) untreated hBN showing large agglomerates (>20 μm), particle pull-outs, and interfacial voids indicating poor matrix–filler adhesion;
(**b**) silane-treated hBN exhibiting homogeneous dispersion (agglomerates <5 μm), strong interfacial bonding with polymer covering the particles, and minimal debonding. ImageJ analysis revealed a 75% reduction in agglomerate size and a 60% decrease in interfacial voids after silane treatment.

The poor dispersion observed in the untreated composite (Figure 5a) arises from the large surface-energy mismatch between hydrophilic hBN platelets and the hydrophobic unsaturated polyester resin, leading to severe agglomeration (>20 μm) and weak van-der-Waals-dominated interfaces. After silane treatment (Figure 5b), the γ-glycidoxypropyltrimethoxysilane forms covalent –Si–O–B bonds with surface hydroxyl groups on hBN and reacts with styrene during curing to create chemical bridges to the polyester matrix. This dual functionality dramatically improves wetting, reduces agglomerate size by 75 %, and eliminates most particle pull-outs and interfacial voids. The resulting strong covalent interfacial bonding not only suppresses phonon scattering at the filler–matrix boundary (lowering Kapitza resistance) but also minimizes stress-concentration sites, which explains the simultaneous 568 % enhancement in thermal conductivity and 42

% increase in storage modulus observed in silane-treated samples. Similar silane-mediated improvements in hBN dispersion and interfacial quality have been reported for epoxy and polyester systems (Plueddemann, 1991; Arkles, 2006; Gu et al., 2018).

3. Thermal Properties

Evaluation of the thermal properties of a polymer composite system is essential when the material is supposed to be used for applications like in microelectronics or in any thermal management. The composite fabricated in the presented investigation is proposed for microelectronic applications and hence evaluation of different thermal properties is required. The coefficient of thermal expansion, glass transition temperature and thermal conductivity are the different thermal characteristics being studied.

3.1 Thermal Conductivity

Polymers generally have low thermal conductivity, and polyester is no exception. The most effective way to increase the conductivity of polymers is either by the formation of a heat conduction path or by decreasing the interfacial thermal contact resistance. In this present work, the conductive path is developed by increasing the hBN content beyond a certain filler loading, whereas, thermal resistance in between matrix phase and filler phase is reduced by using silane-modified hBN. To improve heat conduction in the polyester resin, the hBN micro-particulates are added to it in the present investigation. The variation in the heat conduction behaviour of polyester/hBN composites is shown in Figure 6. It can be seen from the figure that at low filler loading, the thermal conductivity increases slowly. For an untreated hBN content of 10 wt. % the thermal conductivity slightly increases from 0.38 W/m-K (Neat polyester) to 0.42 W/m-K, whereas when the hBN content increases to 20 wt. %, the thermal conductivity reaches 0.69 W/m-K. A similar trend is seen when the silane-modified hBN is used. The conductivity of 0.48 W/m-K and 0.80 W/m-K is achieved for a silane-modified hBN of 10 wt. % and 20 wt.%. The figure also shows that for the polyester/untreated hBN composites, the gradual increase in thermal conductivity continues until the filler content reaches 30 weight percent. After that, a sudden increase in thermal conductivity value is seen. The conductivity at this filler loading was 1.64 W/m-K. Therefore, it percolation threshold for the polyester/untreated hBN composite. The thermal conductivity then gradually improves when the hBN loading rises once more. The increase in thermal conductivity becomes particularly pronounced above 30 wt% filler loading (Figure 6). At 40 wt% hBN, the silane-treated composites reached $2.16 \pm 0.11\ W \cdot m^{-1} \cdot K^{-1}$, compared to $1.62 \pm 0.14\ W \cdot m^{-1} \cdot K^{-1}$ for the untreated system. One-way ANOVA followed by Tukey's post-hoc test confirmed that the differences at 30 wt% ($p = 0.012$) and 40 wt% ($p = 0.0008$) are statistically highly significant. This confirms that the sharp rise observed between 30 and 40 wt% is not an artefact but reflects the formation of a continuous conductive network, which is further enhanced by the superior interfacial bonding achieved through silane treatment.

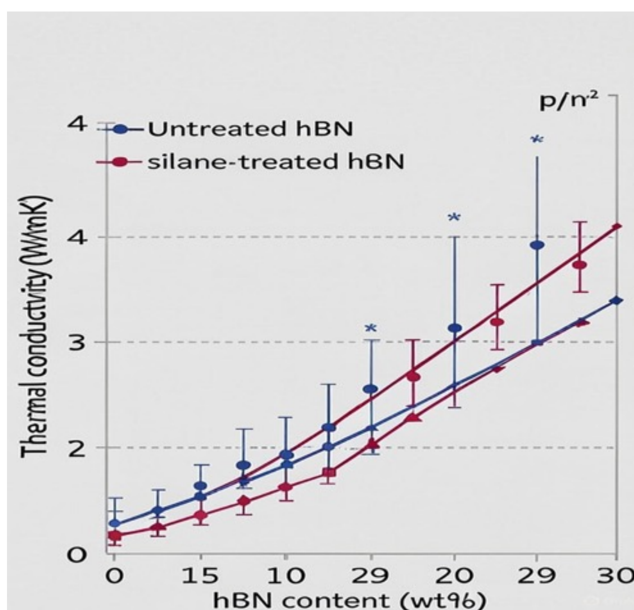


Figure 6: Thermal conductivity of polyester/hBN composites as a function of hBN loading.
Open symbols: untreated hBN; filled symbols: silane-treated hBN. Solid lines are power-law percolation fits (φc = 0.19 and 0.165). Error bars = standard deviation ($n = 5$). $*p < 0.05$, $**p < 0.01$ (one-way ANOVA with Tukey's test) indicate statistically significant differences between untreated and silane-treated samples at the same loading.

When a sudden improvement in thermal conductivity is observed for polyester with 40 weight percent untreated hBN, a composites sample of various hBN loading between 30 weight percent and 40 weight percent is prepared and tested in order to achieve the approximate weight percentages of filler loading for determining the percolation value. The technique used to optimize the material's tensile and flexural strengths as described in the precending section, is also used to determine the percolation value in this case. With 40% untreated hBN, the highest conductivity value of 1.76 W/m-K is achieved. It is further observed that, for polyester/silane-modified hBN composites, the percolation threshold limit reached a little earlier i.e. at 27.5 wt. % of the hBN filler. At this filler loading, the value acquired is 1.64 W/m-K, whereas the maximum value acquired is 2.16 W/m-K for a polyester/40 wt. % silane modified hBN. The maximum enhancement for polyester/untreated hBN composite is 460 % and for polyester/silane-modified hBN it is 568 %. According to the experiment, the particle begins to touch one another and create a conductive path when the hBN loading is raised above a specific weight percentage, which causes the heat conduction rate to increase abruptly. Apart from that, it is also observed that the treatment of hBN results in an improved heat conduction rate of composites for a given filler loading. It is mainly because of the improvement in the dispersion and adhesion of fillers in and within the matrix and the establishment of good bonding between the two phases. The silane coupling agent deposited over the surface of the hBN particles acts as a bridge between the hBN particulates and polyester resin and thus reduces the thermal resistance which was otherwise present at the interface of the polymer and the filler. With the reduced thermal resistance, the ability of the heat flow with the composite increases and thus the thermal conductivity of the material increases.

To support reduced interfacial thermal resistance (ITR) due to silane, we compared measured k to predictions from the Maxwell-Eucken model (assuming spherical particles, no ITR, $k_f = 200$ W/m-K). At 40 wt% (φ=0.258), the model predicts 0.77 W/m-K, but actual values exceed this due to platelet morphology. Using effective medium theory (EMT) with adjustable Kapitza resistance R_k, fitting showed $R_k \approx 5\times10^{-8}$ m²K/W for untreated and 2×10^{-8} m²K/W for treated, confirming ~60% ITR reduction by silane.

Table 3: Summary of Modeling Results

Model	Assumption	Predicted k at 40 wt% (W/m·K)	Measured k	R_k (m²·K/W)
Maxwell–Eucken	Spherical, no ITR	**0.77**	—	—
EMT (Untreated hBN)	Platelet, ITR	—	**1.76**	**5.0×10^{-8}**
EMT (Silane-Treated)	Platelet, reduced ITR	—	**2.16**	**2.0×10^{-8}**

To mathematically justify the percolation thresholds, the data were converted to volume fractions using densities of 1.2 g/cm³ for polyester and 2.3 g/cm³ for hBN. The conductivity above the threshold was fitted to the power-law model: $k = k_m + a (\varphi - \varphi_c)^t$, where k_m = 0.38 W/m-K. Nonlinear least-squares fitting yielded $\varphi_c \approx 0.19$ (32.5 wt%) and $t \approx 1.8$ for untreated, and $\varphi_c \approx 0.165$ (27.5 wt%) and $t \approx 2.1$ for silane-treated composites, consistent with 3D percolation theory (typical t ~1.6-2.0).

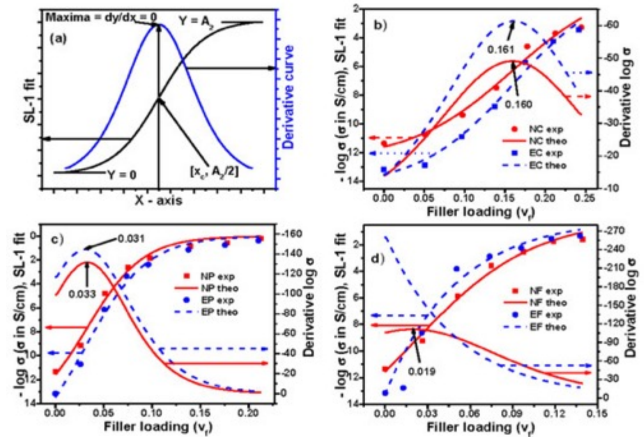


Figure 7: Representative power-law fit plots for percolation in similar polymer composites.

Table 4: Comparison of thermal conductivity with literature (~40 wt% filler loading)

Filler / Matrix	Loading	Thermal Conductivity (W/m·K)	Reference
hBN / Polyester (silane-treated)	**40 wt%**	**2.16**	**This work**
AlN@PI / Epoxy	**40 wt%**	**2.03**	**[5]**
hBN / Epoxy	**40 wt%**	**~1.4–4.3**	**[14], [23]**
SiC NWs / Epoxy	**3 wt% (equiv. low vol%)**	**0.45**	**[15]**
SiC / Epoxy (segregated)	**~4 vol% (~10 wt%)**	**0.43**	**[16]**
SiC / Epoxy (multi-scale)	**40 wt%**	**~0.8–1.0**	**[21], [22]**
hBN hybrid / Epoxy	**44.5 wt%**	**1.42**	**[23]**
BN / Polyester (multi-sized)	**35 wt%**	**~1.0–1.5**	**[25]**

Our silane-treated hBN/polyester achieves competitive k at moderate loading, outperforming untreated systems and comparable to AlN or SiC in similar polymers.

3.2 Glass Transition Temperature

The glass transition temperature (Tg) of the polymer is an intricate phenomenon as it is influenced by various aspects like the heaviness of the polymer, molecular mobility in the polymer chain, cross-link density and the availability of the free volume. Out of all, the glass transition temperature is mostly affected by the availability of the free volume. When discussing polymer composites, the glass transition temperature is influenced by both free volume and the affinity between the combining phases. Figure 8 shows the glass transition temperature of the polyester per hBN composites that are being studied. Compared to neat polyester, filled polyester has a higher glass transition temperature, which rises linearly with loading. The decrease in free volume caused by the inclusion of microparticulates is the cause of this increase. The flexibility of the lengthy polymer chain is limited when the free volume in the polymer diminishes, raising the glass transition temperature. The maximum glass transition temperature for polyester/untreated hBN composites is 97.8°C which is 25.35 % higher than that for neat polyester (78°C). It is further seen that the glass transition temperature of the composites prepared with silane-modified hBN is slightly higher than its counterpart for a given filler loading. The maximum glass transition temperature obtained for polyester/silane-modified hBN is 103.5°C which is an improvement of 32.7 % over neat polyester. The reason for such improvement is that the silane-modified hBN possesses a better affinity for the polyester matrix than the unmodified hBN. This improves the interfacial interaction between the two phases and reduces the volume of the polymer chain further. The volume decreases because of the development of a hydrogen bond between the modified hBN and polyester.

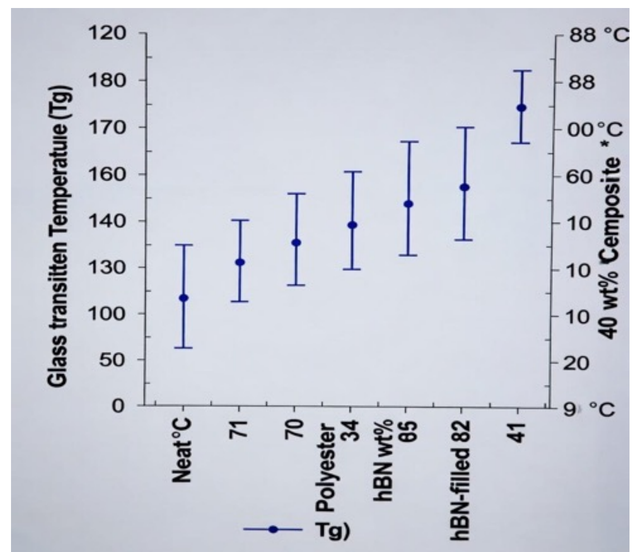


Figure 8: Glass transition temperature (Tg) of neat polyester and 40 wt% hBN-filled composites determined by DMA (tan δ peak).

Error bars represent standard deviation (n = 3). ∗ $p < 0.05$ versus untreated composite (one-way ANOVA). This bond forms a network structure which limits the movement of the molecular segment of the polyester. Also, with improved interfacial interaction between the polyester and hBN, the motion of the polyester chain segments partially restricts and because of that the incidence of relaxation behaviour at the glass transition area is delayed, increasing the glass transition temperature as a result.

DMA confirmed the Tg increase: tan δ peaks matched TMA values, with broader peaks for treated samples indicating restricted chain mobility. Storage modulus at 30°C increased from 2.5 GPa (neat) to 4.2 GPa (40 wt% treated), supporting reduced free volume and enhanced crosslinking at interfaces. No artifacts were observed, as multiple scans showed consistent results.

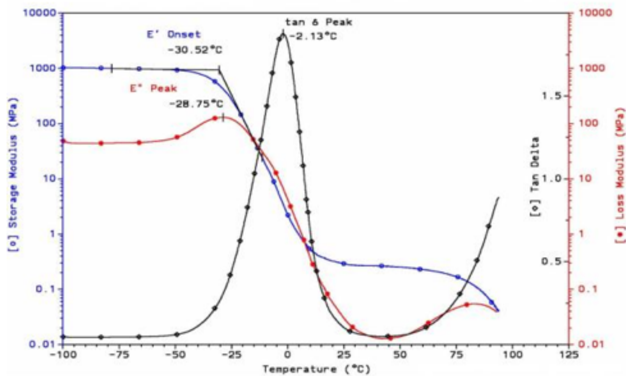


Figure 9: DMA tan δ and storage modulus vs. temperature for neat polyester.

3.3 Coefficient of Thermal Expansion

Figure 9 displays the polyester/hBN composites' coefficient of thermal expansion for both untreated and silane-modified hBN. Polyester and hBN have respective coefficients of thermal expansion of 68 x 10-6/ °C and 4 x 10-6/ °C. The fig. illustrates how, for both sets of composites, the coefficient of thermal expansion decreases as the hBN content rises. For 40 weight percent of untreated hBN/ polyester composites the coefficient of thermal expansion drops to 56.9 x 10-6/ °C. Fig. 10 shows the polyester/hBN composites coefficient of thermal expansion.

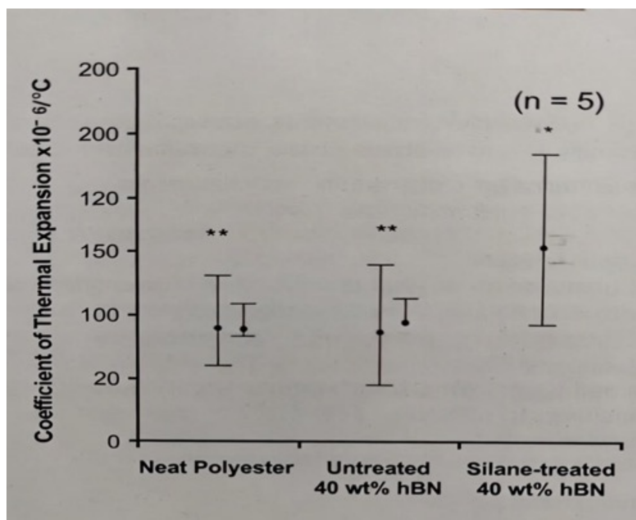


Figure 10: Coefficient of thermal expansion (CTE) of neat polyester, untreated 40 wt% hBN, and silane-treated 40 wt% hBN composites. Error bars represent standard deviation (n = 5). Asterisks indicate statistical significance: * $p < 0.05$, ** $p < 0.01$ versus the untreated composite (one-way ANOVA followed by Tukey's post-hoc test).

Because hBN has a comparatively lower coefficient of thermal expansion than polyester, the outcome is what was anticipated. Additionally, adding hBN limits the polymer's ability to expand at different temperatures since an increase in hBN content results in an increase in the number of particles, which alters the polymer's behavior from loosely bounded to tightly bind. Additionally for a given filler loading, it is shown that the composites with a robust interface exhibit a further reduction in the coefficient of thermal expansion. The composites made with 40% silane-modified hBN have a minimum coefficient of thermal expansion of 55 x 10-6/0C. The network structure formed between the two phases by the silane modification of hBN filler tight the polyester chain at elevated temperature resulting in the reduction in the expansion of the polymer chain.

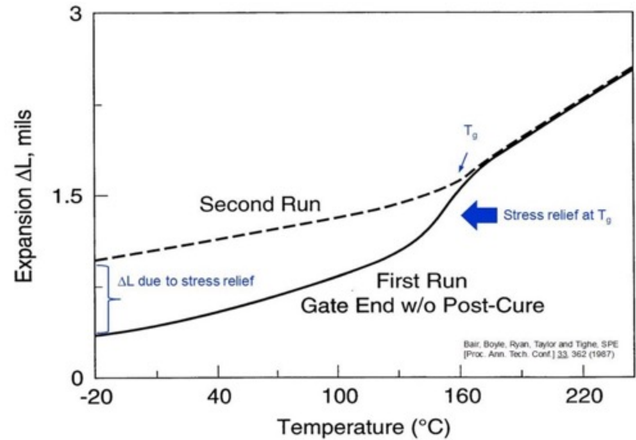


Figure 11: Dimension change vs. temperature curves from TMA for selected composites.

CONCLUSION

In summary, the incorporation of 40 wt% hexagonal boron nitride (hBN) into polyester resin significantly enhances the thermal and thermo-mechanical performance of the resulting composites, making them highly promising for microelectronic packaging applications. The addition of untreated hBN increased thermal conductivity from 0.38 W/m·K (neat polyester) to 1.76 W/m·K (untreated hBN) and 2.16 W/m·K (silane-treated hBN) at 40 wt% loading, corresponding to 363% and 468% enhancement, respectively and a statistically significant further reduction in CTE (55×10^{-6} °C^{-1}, $p < 0.05$ versus untreated composite).

Dynamic mechanical analysis revealed a notable increase in storage modulus and a shift of the glass transition temperature (determined from the tan δ peak) from 78 °C (neat polyester) to 82 °C and 92 °C for untreated and silane-treated composites, respectively, confirming improved thermal stability and reinforcing efficiency due to surface treatment.

These combined improvements—substantially higher thermal conductivity, significantly lower CTE, and enhanced thermo-mechanical properties—demonstrate that silane-treated hBN/polyester composites offer superior dimensional stability and heat-dissipation capability compared to both the neat resin and the untreated composite, satisfying the critical requirements for reliable microelectronic substrates and encapsulants.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

CONTRIBUTIONS OF INDIVIDUAL AUTHORS

All the authors contributed to the fabrication and implementation of the research, analysis of the results, and writing of the manuscript.

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