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Novel high temperature polymeric encapsulation material for extreme environment electronics packaging

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HIGHLIGHTS

- Resorcinolbased phthalonitrile (rPN) has been developed as a harsh environmentelectronics encapsulation material.
- Hybrid polymer of rPN is tunable to havecomparable thermal, mechanical and electrical properties as epoxy molding compound.
- The rPN hybrid polymer can beintegrated onto dual-in-line packages which withstood extreme environment of 310°C at 190 MPa.

GRAPHICAL ABSTRACT
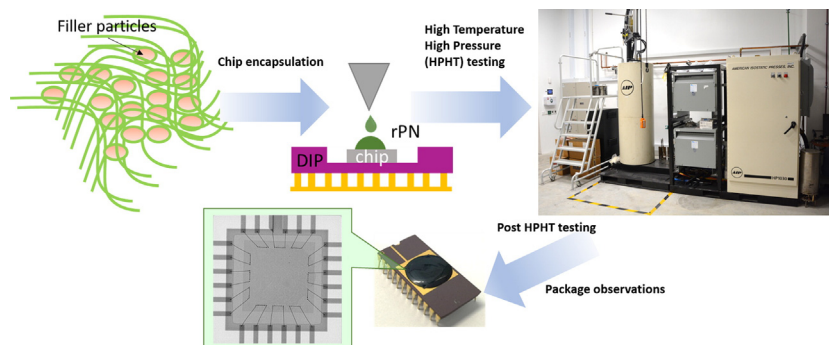
ARTICLE INFO

ABSTRACT

This study explores the usage of resorcinol based phthalonitrile (rPN) in harsh environment electronics encapsulation applications. rPN itself exhibits excellent properties as a high temperature polymeric molding compound in terms of mechanical properties and thermal stability. Its properties improve with thermal aging, outperforming other traditional polymers at operational temperatures close to 300 °C. Optimal bond shear strength of rPN is achieved when used as a monomer or pre-polymer with a low melting point of 180 °C, which is compatible with today's electronic packaging processes. The hybrid polymer of rPN with fillers, such as silica or alumina, has a coefficient of thermal expansion (CTE) which is highly tunable, allowing the rPN to have strong adhesion to the underlying substrates and chips. The properties of the rPN hybrid polymer is the result of strong bond interactions between rPN and the fillers, as verified by Fourier Transform Infrared Spectroscopy (FTIR) and Density Functional Theory (DFT) studies. We further demonstrate the integration of the rPN hybrid polymer onto dual-in-line packages (DIPs), which did not fail when subjected to an extreme environment of 310 °C at 190 MPa. This new polymer matrix composite may thus revolutionize the existing thermal-mechanical limits of plastic electronics packaging for extreme environment.

1. Introduction

Advances in the field of oil drilling [1–5] and geothermal energy [6–8] extraction have made it possible to tap into previously uneconomic energy reserves that are hard to reach, such as crude oil reservoirs in

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deep sea locations or geothermal energy reserves stored at temperature around 300 °C and pressure above 200 MPa [9,10]. Finding and accessing these untouched reserves involve operating in increasingly harsh conditions, as well as ensuring the survivability of delicate electronic components in order to deliver high-quality measurements reliably. The increasing use of advanced recovery techniques has thus dramatically increased the materials' requirements for high pressure and high temperature (HPHT) technology.

Typically, ceramic encapsulation comprises high temperature bonding materials such as glass frit which requires processing temperature of around 400 °C [11] to achieve hermetic sealing for electronic component protection. However, such a high temperature may damage the sensitive electronic components enclosed. In addition, the ceramic encapsulation contributes to the overall weight of the whole component. The demand for lightweight, high performance materials capable of withstanding higher temperature and pressure increases exponentially with the drill depth and has led to an upsurge in the development of easily processable encapsulation materials.

Polymer-based encapsulations are not widely used in high temperature applications due to its inferior thermal behavior as compared to ceramic counterparts. The current state-of-the-art epoxy does not allow substantial improvements for application temperatures above 300 °C. Amongst the high temperature resistant polymer resins, cyanate esters [12–15], polyimide [16–19], and bismaleimide (BMI)-based composites [20–23] have been widely investigated in recent years. Cyanate ester [24] has emerged as the most appropriate candidate as it can withstand temperatures of about 250 °C. However, cracking under high temperature remains an unsolved issue. Despite having a good thermal stability, the high moisture absorption nature of polyimide makes its adoption as an encapsulation material an issue due to reliability concerns, especially in areas of high moisture content. On the other hand, a short high temperature dwell test has revealed that current resins such as epoxy and BMI are also unsuitable for operations at temperatures above 300 °C. As shown in Fig. 1, both epoxy and BMI [25] samples decomposed within 1 h of high temperature soaking at 300 °C.

Phthalonitrile (PN) was identified as a high temperature resistant polymer and significant research efforts have been placed on the synthesis and characterization of phthalonitrile [26–29], its derivatives [29,30] and composites [31,32]. Though the potential of PN as an electronics packaging material has been postulated, no one has yet demonstrated such an application to the best of our knowledge, especially its survivability under HPHT conditions. Resorcinol-based phthalonitrile (rPN) is well known as a high-temperature thermoset with low moisture sensitivity of typically <1% [33]. Its relatively low melting temperature of 183 °C and curing profiles, coupled with its excellent tunable thermal and mechanical properties, make rPN a novel modifiable polymer material for electronics packaging, which can widen the potential applications of the polymer and drilling capabilities in the HPHT application regime.

Thermal management is a crucial factor enabling the use of polymeric encapsulation materials under high temperature applications. The large coefficient of thermal expansion (CTE) mismatch between the silicon chip and the organic encapsulant severely diminishes the reliability of the electronic device involved. Current approach used in countering the CTE mismatch is by filling the encapsulant with ceramic fillers to reduce the stresses arising from the differences in the CTE between the encapsulant, the substrate and the chip. From our study [32], PN composites of silica or alumina fillers may be an alternative as a new class of high performance polymer-based encapsulant for HPHT applications. The advantage of these hybrid organic-inorganic materials is that the fillers —OH and —CN functional groups interact chemically with the matrix to withstand the extreme environment. Furthermore, the addition of alumina or silica improves the compatibility of the CTE with the substrate and chip. Lastly, the high compressive strength of rPN [34] can protect the encapsulated electronics in a high pressure environment.
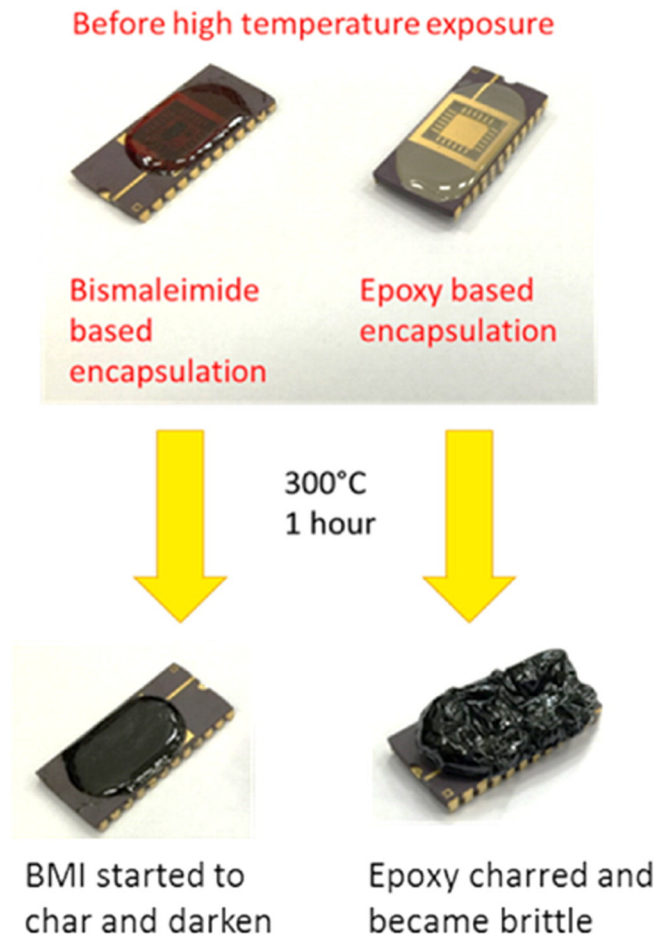

Fig. 1. Demonstration of BMI and epoxy based encapsulation survivability.

In this paper, we investigate the effects of silica and alumina fillers addition to rPN on its mechanical, thermal and electrical properties. We also demonstrate the suitability of rPN based resins as an electronics package encapsulation for an extreme environment where the temperature exceeds 300 °C and pressure reaches 190 MPa.
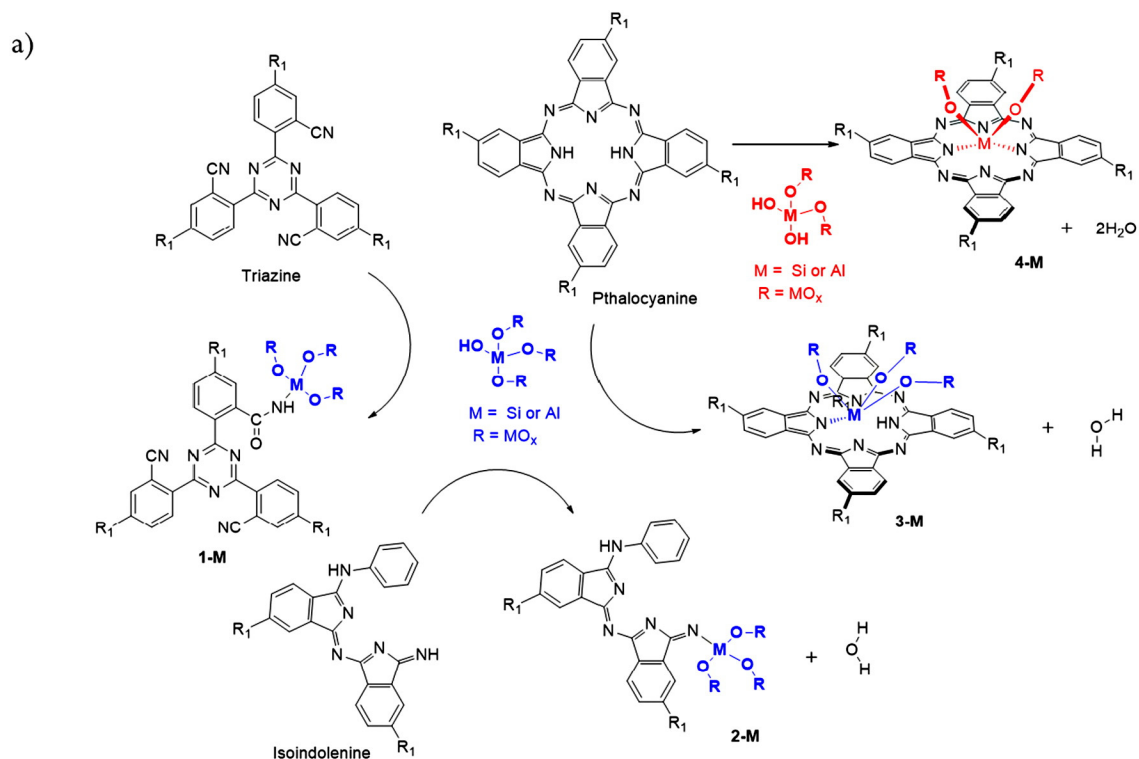
2. Experimental

2.1. Materials

4-Nitrophthalonitrile (98.0%) was supplied by ANR Technologies. Dimethyl sulfoxide (DMSO), potassium carbonate (>99.0%) and silica particles were purchased from Sigma Aldrich. 1,4-bis(4-aminophenoxy)benzene (p-APB) was purchased from Tokyo Chemical Industry (TCI). Alumina particles were purchased from International Laboratory, USA. The silica particle size distribution was from 0.5–10 μm (approximately 80% between 1 and 5 μm) while alumina particles were mainly in the size range of 0.5–2.5 μm. All chemicals were used as received. Bis(3,4-dicyanophenoxy)benzene or resorcinol-based phthalonitrile (rPN) was synthesized using a modified procedure reported by Keller et al. [27].

2.2. Preparation of rPN composites

The silica or alumina fillers were mechanically mixed with molten rPN at 200 °C for 2 h before 2.5 mol% of p-APB was added and mixed for another 10 min. A sudden quench was used to freeze the polymerization process temporarily. The dark green mixture was then pulverized into powder and pressed into pellets, similar to those made from epoxy molding compounds that are used in transfer molding. The pellets can

Fig. 2. (a) Possible pathway of rPN polymerization product with silica and alumina fillers. R1 represents the rest of the polymeric chain in the actual product. (b) Change in enthalpy for the truncated structures in the DFT calculations.
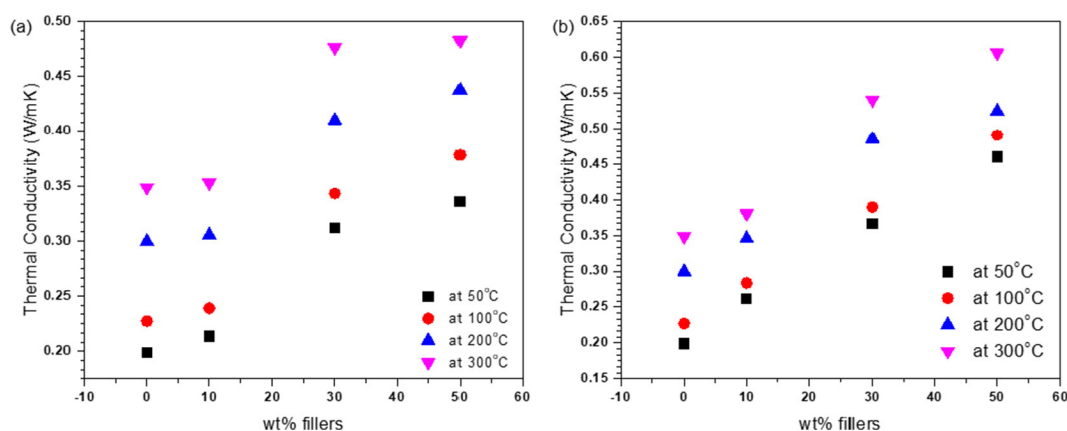
Fig. 3. Thermal conductivity of rPN composites as a function of (a) silica and (b) alumina filler wt% at different temperatures.

be re-melted at 180 °C and is low enough not to damage the underlying metallic wire-bonds and silicon chip.

2.3. Density Functional Theory (DFT) studies on the interaction between rPN and fillers

In order to correlate the physical properties of the rPN-filler composites with their chemical structure, the chemical bonds that form between the silica or alumina fillers and the macromolecules of rPN during polymerization were theoretically optimized using the ωB97x-D density functional method [35] coupled with the 6-31G** basis set. The ωB97x-D functional method was chosen as this empirical function is better suited to the system than normal hybrid DFT methods (e.g. B3LYP) [36,37] in handling kinetics, thermodynamics, and non-covalent interactions. Frequency analyses were performed on all ωB97x-D/6-31G** optimized geometries to confirm the nature of the stationary points as minima structures (with all real frequencies). Single point calculation with ωB97x-D/6-311 + G** based on the gas-phase ωB97x-D/6-31G* optimized geometry was taken for the relative enthalpy energies reported. All calculations were performed using the Gaussian 09 suite of programs.

2.4. Characterization

Chemical bond studies were conducted using Perkin Elmers Frontier FTIR to determine the new bond formation between aluminum or silicon atoms from the fillers to nitrogen atoms within the polymer. Flexural modulus was measured with Instron 5567 equipped with a 30 kN load cell with deflection rate set to 0.5 mm/min. For compressive strength measurements, Instron 8516 equipped with 100 kN load cell

was used. Samples were compressed at 3 mm/min until they cracked. To determine the elastic modulus, a Triboindenter TI-950 (Hysitron, MN, USA) was used. The machine was equipped with a standard 2D transducer and a cube corner tip, which was calibrated with a standard fused quartz sample for the required contact depth. A load function with a 5 s-2 s-5 s loading-holding-unloading time and a maximum load 500 μN was used. Oliver-Pharr method was used to calculate the elastic modulus of the sample over the unloading cycles. Ten indentations were performed on each sample. Bond shear testing was performed using a DAGE 4000 shear tester with a 100 N load cell at a shearing rate of 50 μm/s and a shear height of 150 μm was set at the start of every shear repeat. Linseis XFA 500 with a calibrated polyimide reference was used to determine the thermal conductivity of the sample. The choice of reference is especially important as both rPN and polyimide have relatively low thermal conductivity as compared to ceramic and metal samples. The CTE was measured using TMA Q400 from TA Instruments in inert nitrogen ambient; samples were heated at a rate of 10 °C/min. The dielectric constant was measured using Agilent 4284A LCR meter at 1 MHz. The cross-sectioned samples were observed under the field emission scanning electronic microscope (FESEM, FEI Nova 600i). X-ray analysis was performed using Shimadzu CT scanner with a voltage of 70–100 kV and a current of 60–90 μA.

3. Results and discussion

3.1. DFT studies on the interaction between rPN and fillers

The structures of alumina and silica cluster systems were used for the DFT model to incorporate the 3-dimensional steric effects that the polymer may encounter when interacting with the fillers. Truncated
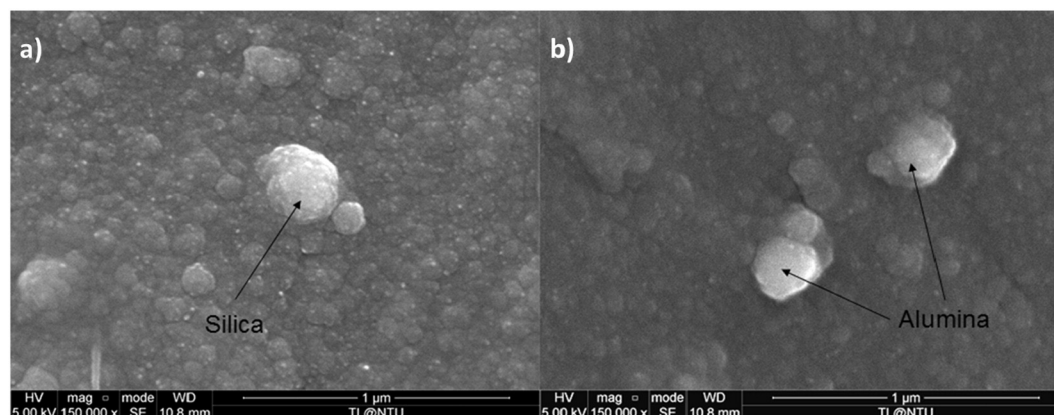

Fig. 4. SEI images of 30 wt% (a) silica filled rPN and (b) alumina filled rPN.
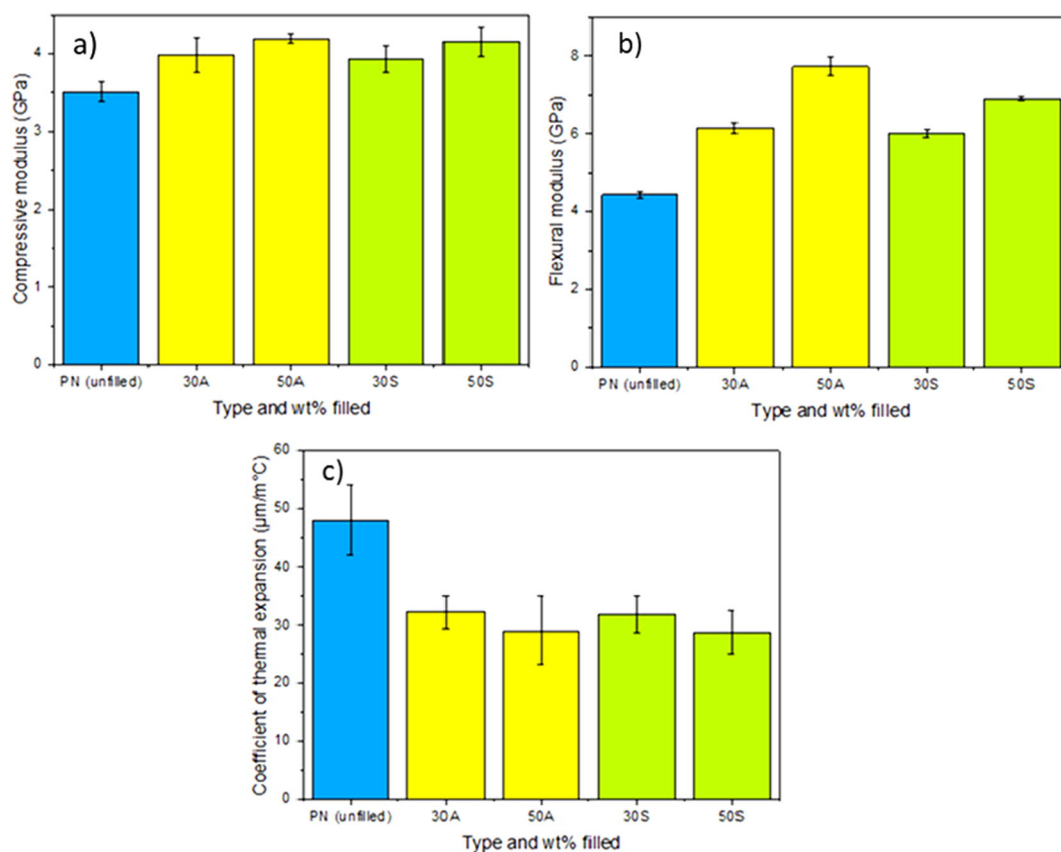
Fig. 5. (a) Compressive modulus, (b) Flexural modulus and (c) CTE of rPN matrix as a function of filler composition; A: alumina, S: silica.

structures of the polymer products were used for the DFT calculation as shown in Fig. 2(a). The enthalpy of formation of adducts illustrates the thermodynamic feasibility of the reaction, with a negative value indicating a favorable bonding mechanism between the fillers and rPN molecular products.

The alumina and silica cluster systems were used for the DFT studies with the truncated polymerization products for rPN. Fig. 2(b) shows the calculated change in enthalpy with respect to the polymerization products and the silica or alumina clusters. Triazine **1-Si** and phthalocyanine **4-Si** covalent bonded adduct to the silica clusters show negative enthalpy values of −108 kJ/mol and −122 kJ/mol, respectively. These results are in tandem to alumina adducts with triazine (**1-Al**) and phthalocyanine (**4-Al**) which show higher negative enthalpy values of −163 kJ/mol and −185 kJ/mol, respectively. Thermodynamically favorable enthalpy of covalent bond formation between the aluminum/silica clusters and the polymerization products correlates well with the FTIR peaks of Si—N and Al—N identified in the formulated silica and alumina rPN composites, respectively (Figs. S1 and S2 in Supporting information). The presence of such new bond formation helps to keep silica and alumina fillers strongly bonded to the matrix. On the other hand, isoindolenine does not form energetically favorable adducts with both silica and alumina adducts as reflected in the high enthalpy

of formation. This is important as fillers which do not form bonds with the matrix may create voids and lead to potential failure under stress subsequently.

3.2. Characterization of rPN-fillers composites

Fig. 3(a) and (b) plot the thermal conductivity of alumina-rPN and silica-rPN composites as a function of filler percentage, respectively. The thermal conductivity of rPN composites increases with increasing wt% of fillers due to better heat transfer in the composites with fillers being closer together as silica and alumina possess higher thermal conductivity as compared to pure rPN. The thermal conductivity increased from 0.20 W/m-K for pure rPN resin to 0.48 W/m-K (alumina/rPN) and 0.32 W/m-K (silica/rPN), with 50 wt% fillers at 50 °C. Alumina/rPN composite shows a better improvement in the thermal conductivity as compared to silica/rPN composite at the same wt% loading and temperature due to the intrinsically higher thermal conductivity of alumina particles. The thermal conductivity values are comparable to that of epoxy molding compound where the conductivity value ranges between 0.6 W/m-K to 1.34 W/m-K [25], but typically with high weight compositions of fillers of up to 90 wt%. The rPN matrix has thus effectively reduced the amount of ceramic fillers needed to obtain the same degree of improvement in thermal conductivity.

It is important to achieve a uniform dispersion of the fillers in the matrix since the mechanical and thermal properties of composites are closely related to the dispersion and adhesion of the particles with the matrix. SEI images were taken for the composites before the properties were characterized and Fig. 4 shows the cross-section SEI micrographs of the facture surface of rPN with 30 wt% silica and alumina particles. It is observed that the fillers, which appear brighter against the darker polymer matrix, are well dispersed in both samples, with no large clusters detected. In comparison, epoxy molding compound are typically loaded up to 90 wt% with fillers. The SEI images also reveal good

Table 1
Dielectric constants for silica and alumina filled rPN composites.

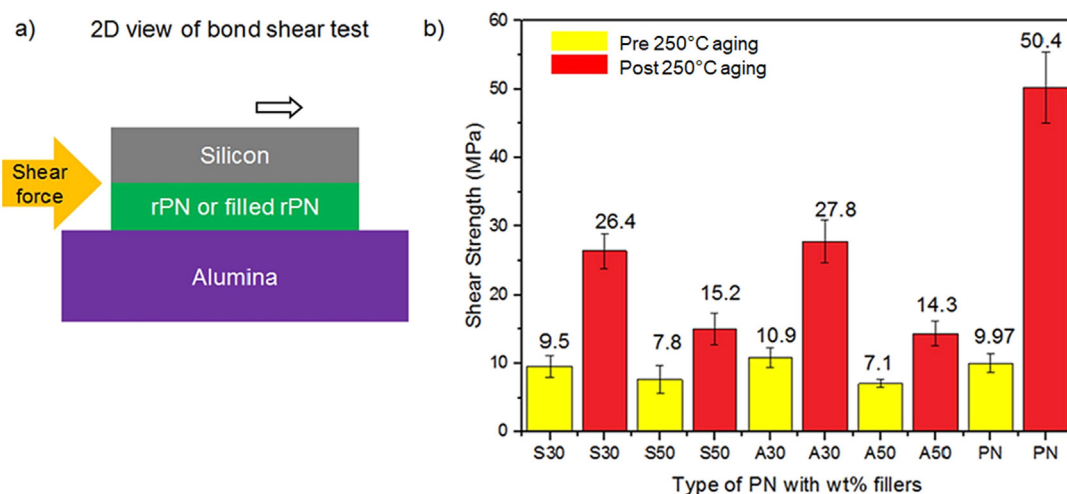| Filler material types	Fill (wt%)	Dielectric constant
rPN	0	7.36
Silica	10	6.22
	30	5.34
	50	3.99
Alumina	10	6.44
	30	6.76
	50	7.66

Fig. 6. (a) Schematics for material stack for bond shear testing. (b) Bond shear strength of rPN based composites before and after thermal aging.

bonding between the fillers and matrix, which implies good stress transference between rPN matrix and its fillers. This may also explain the high thermal conductivity values of the composites shown earlier.

The mechanical properties of the rPN composites are shown in Fig. 5. The compressive modulus varies with the filler wt%, in the range of 3.5–4.2 GPa as shown in Fig. 5(a). This is comparable to commercial epoxies, where its compressive modulus typically ranges around 1.0–35 GPa [25], which are highly filled with fillers. Flexural modulus are measured to be around 4.4–7.7 GPa, as shown in Figs. S4 and 5(b), respectively. These are also well in the range of 2.4–32 GPa in comparison with flexural modulus of commercial epoxy molding compound [25]. It can also be observed that alumina filled rPN composites are stronger than silica filled ones. This is because the composite modulus is dependent on the compressive modulus of the fillers. The rule of mixtures can be used to predict the trend in the compressive modulus but an over-prediction is expected of the resulting composite because it assumes conditions of bonding ideality. During cross-linking, some polymer chains may not align perfectly

for bond formation between the matrix and the fillers. Nevertheless, the measured modulus does increase with higher wt% of fillers added and the increase is also higher for alumina over silica ones. This is in agreement with the simulated change in enthalpy values shown in Fig. 2(b), where alumina fillers consistently show more negative enthalpy energies when bonded with triazine and phthalocyanine than silica fillers.

On the other hand, an increase in fillers content significantly lowers the CTE of the rPN composites as shown in Fig. 5(c). It is therefore possible to match the CTE of rPN composite with both substrate and die materials. Addition of 50% fillers can help to reduce the CTE of rPN from 48 μm/m °C to about 29 μm/m °C for both filler types, in comparison with epoxy molding compound which has CTE in the range of 3.3–80 μm/m °C, and compatible with the materials to be encapsulated such as the silicon die (2.6 μm/m °C), alumina substrate (8.1 μm/m °C) and gold wirebonds (14 μm/m °C).

Besides mechanical properties, electrical properties such as dielectric constant are crucial considerations for rPN to be adopted as an
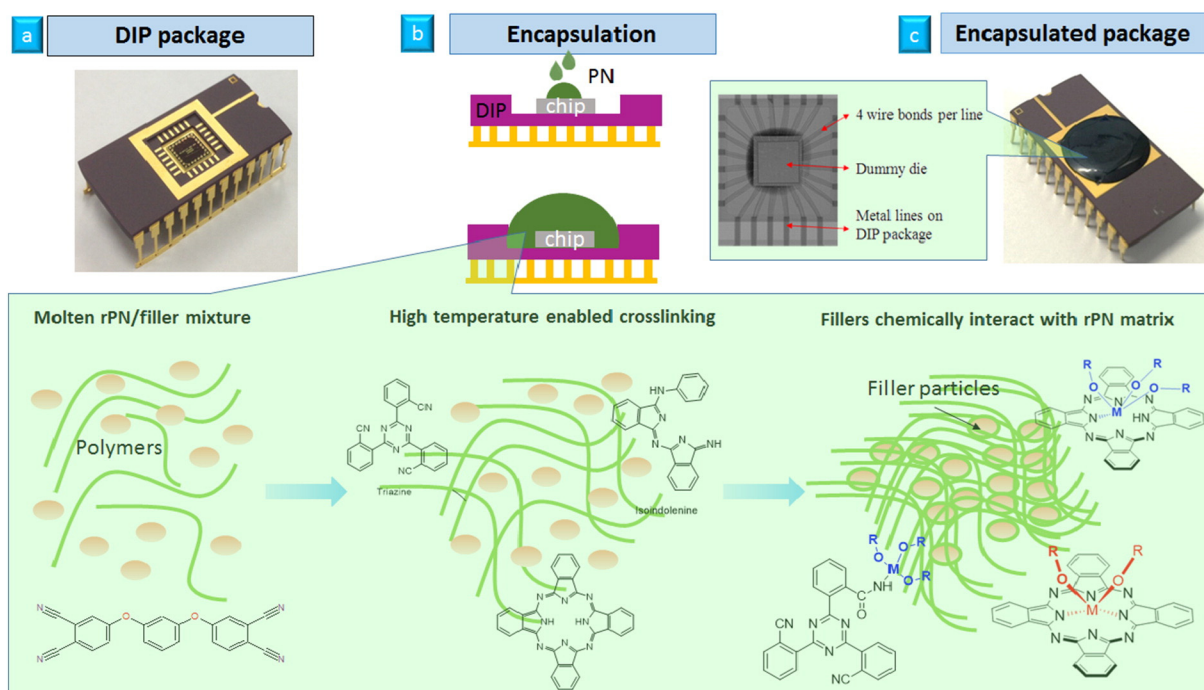

Fig. 7. (a) DIP with chip bonded. (b) Encapsulation process of DIP by molten rPN-filler mixture where M denotes Al or Si and R represents the repeating oxide groups. (c) Encapsulated package with an X-ray image of the wire-bonds.

encapsulant to reduce cross-talk and signal propagation delay. Table 1 shows the dielectric values of rPN with various amount of fillers added. The dielectric constant measured ranges between 7.36 for neat rPN to 3.99 when 50 wt% of silica is added. As silica has a lower dielectric constant of 3.8, this effectively lowers the overall dielectric constant as a composite. The dielectric constant for rPN with alumina filler is higher than that with silica filler for all the same weight percentage added. The higher measured dielectric constant is due to the higher dielectric constant of alumina at 9.8. A decrease in the dielectric constant of rPN with low wt% of alumina fillers is likely due to air bubbles introduced during the mixing. Typically, epoxy molding compound has a dielectric constant of 3.13 to 7 [25]. This shows that silica filled rPN composites can also be tuned to be comparable to the dielectric constants of epoxy molding compound.

Adhesion and cohesion properties are important when composites are used as an encapsulant or die attach material. Fig. 6(a) shows the test structure design for evaluating the bond strength of rPN composites when utilized as a die attach for bonding silicon chips to high temperature resistant substrates such as ceramic (i.e. alumina). Fig. 6(b) shows the bond shear strength of rPN based composites before and after thermal aging at 250 °C for 250 h, which mimics extreme drilling conditions especially in the regime of HPHT [38]. In general before thermal aging, the addition of filler particles slightly decreased the bond strength of the composite and the decrease became more apparent when the filler loading was increased to 50 wt%. The sheared samples typically show that failures occurred at the Si/rPN interface, with some mixed mode failures as well.

On the other hand, thermal aging led to a drastic increase of the shear strength. The bond strength of pure rPN resin improved by 5 times while smaller improvements were observed for the composites.

This increase is likely due to further cross-linking of the thermoset rPN between the polymeric chains and also increased interaction of the resin with the filler atoms, enhancing its strength during high-temperature storage. This increase in strength is advantageous for rPN both as a die attach and encapsulant material.

The increase in strength for die attach however is not directly proportional to the increase in filler weight percentage, as a decrease in shear strength is observed under high filler wt% content. This can be attributed to the formation of agglomerates acting as defects, which are known to be detrimental to mechanical properties. Following simulations results shown in Fig. 2b, when rPN is used in the form of a die attach layer, agglomerated fillers are harder to disperse leading to higher chance of stress concentration zones formation. This effect increases the probability of failure and causes the measured results to differ from theoretical expectations. An increase in alumina/silica fillers also means less rPN for bonding within the matrix, hence the decrease in bond shear strength as the filler weight percentage is increased to 50 wt%. Nevertheless, the shear strength of all the compositions exceeds 6.9 MPa, which is the requirement from the MIL-STD-883 Method 2019 standard.

3.3. rPN composites as extreme environment electronics packaging encapsulant material

Fig. 7 illustrates the process flow to integrate rPN composites into electronics packages, such as a dual-in-line package (DIP). Fig. 7(a) shows the DIP with wire-bonds to the silicon chip. The molten rPN-alumina/silica filler mixture was cast into the cavity of the package (Fig. 7(b)) and subjected to a heat treatment. The inset of Fig. 7(b) shows the cross-linking process and predicted chemical interactions between rPN and fillers. In Fig. 7(c), the fully processed rPN-filled DIP with
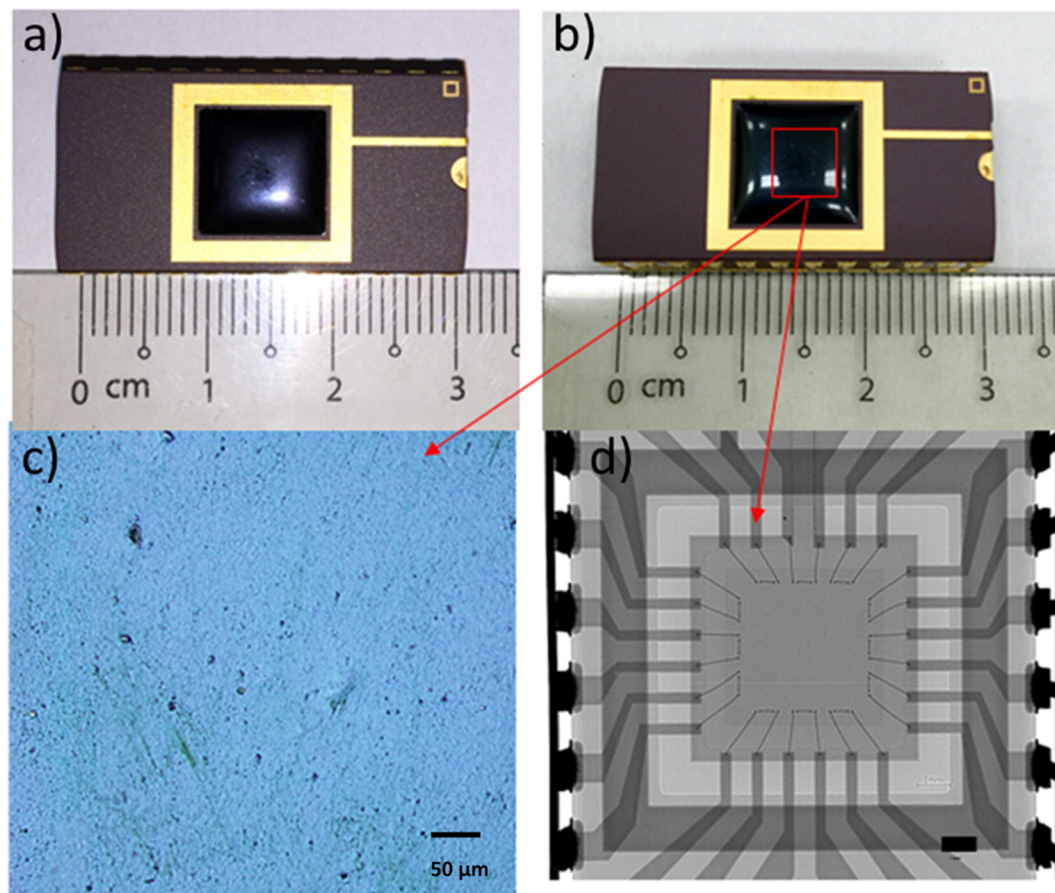

Fig. 8. Images showing the encapsulated DIP (a) before and (b) after being subjected to HPHT conditions. (c) Optical imaging shows no visible cracks on the encapsulated surface. (d) X-ray imaging reveals the wire-bonds remained intact after sample was subjected to 190 MPa at 310 °C.

inset shows the X-ray image of the package. It can be observed that the wire-bonds show no negative wire sweep effect after the encapsulation process due to the low viscosity of the molten rPN-filler mixture.

Using a customized integrated HPHT tester [34], neat PN encapsulated on a ceramic DIP was found to crack under a test condition of 250 °C and 138 MPa. On the other hand, DIP encapsulated with 50 wt% silica filled rPN was found to survive at a more stringent test condition, a maximum of 310 °C and 190 MPa (equivalent to 1875 atm) for a dwell duration of 168 h. From Fig. 8, a crack-free surface was observed for the samples placed under a uniform pressure of 190 MPa, indicating that rPN has the mechanical endurance under such external pressure. No damage was observed on the sample before and after testing using both 2D and 3D computed tomography X-ray inspection. The wire-bonds remained intact as revealed from the X-ray images, likely due to the significantly reduced CTE mismatch between the encapsulant, the substrate and the die that relieved the stresses generated by the thermal expansion which might have damaged the delicate wire-bonds. Pre and post-stressing sample inspections were also performed using SEM with no cracks observed under high magnifications.

4. Conclusions

In this work, we have demonstrated the development of a novel high temperature harsh environment electronics packaging encapsulation material synthesized from resorcinol based phthalonitrile. Through the addition of silica or alumina fillers, the thermal, mechanical and dielectric properties of the rPN can be tuned to the operating environment. The addition of up to 50% of the inorganic fillers greatly reduced the CTE of rPN matrix, enabling it to be molded over Si chips and ceramic substrates without cracking. Its dielectric constant and thermal conductivity values are also in the range of typical electronics encapsulation material. With 50% loading of the fillers, the composite remains processable as demonstrated in the encapsulation of a wire-bonded Si chip on a dual-in-line package. Upon exposure to HPHT testing condition of 310 °C and 190 MPa, the encapsulated DIP remained structurally intact without any detectable damage through both SEM imaging and X-ray analysis. The results demonstrate the outstanding properties of phthalonitrile-based polymer resin as a high-performance polymer encapsulant for electronic packages for extreme conditions.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.matdes.2017.12.029.

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