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Low loss polypropylene-silicon composites for **millimetre** wave applications

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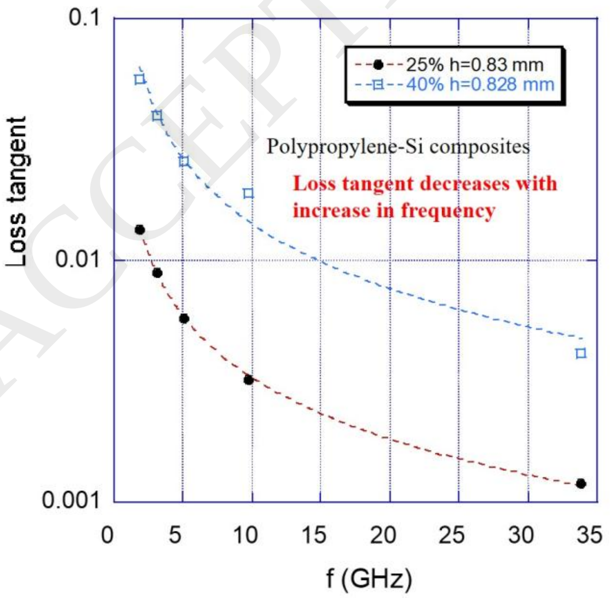
Graphical abstract



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Highlights

- All reported polymer-ceramic composites or hard ceramic substrates for microwave circuit applications show an increase in dielectric loss with increasing frequency in the microwave/millimetre frequency range. This is really problematic for high frequency applications such as IOT and 5G which are at very high frequencies. In the present paper we report for the first time the development of a composite (polypropylene-silicon) which shows a decrease in loss tangent with increasing frequency indicating the possibility of their use in 5G and Internet Of Things (IOT) as well as other high frequency or mm wave applications. This will lead to immense research on polymer-semiconductor composites for high frequency applications. The results will be of great advantage in the future high frequency wireless communication and other applications.

Abstract

Polypropylene-silicon composite laminates **were** fabricated **via** hot pressing and **their** dielectric properties up to 35 GHz **were studied**. The loss tangent **was found to decrease** with increasing frequency whereas an increasing trend of loss tangent **has previously been reported in the literature for such** composites. The decreasing trend of the loss tangent with frequency is due **to the** decreasing conductor loss of silicon (Si)

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and = indicates the possibility **for** use in high frequency applications such as **the** Internet Of Things and **5th** generation wireless communication. The composite containing 25 vol% of Si **had a** relative permittivity of 3.5 and **a** loss tangent of 0.001 at 35 GHz. The coefficient of thermal expansion and **the** thermal conductivity of 40 vol% Si loaded composites **were** 51 ppm/^{o}C and 2.23 W/mK, respectively. The thermal conductivity of the composite increased by 163 % and 913 % for 25 and 40 vol% Si addition, respectively.

Key words: Millimetre wave materials, Microwave materials, Polymer-Silicon composites, Dielectrics, CTE, Thermal conductivity

1. Introduction

Materials with **a** low loss tangent and low relative permittivity (ε_r) are essential for **the** next generation **of millimetre**-wave communications. Recently, millimet**re**-wave wireless communication has begun to spread to public welfare systems in many countries. The frequency of millimet**re** wave communication ranges from 30 GHz (10 mm) to 300 GHz (1 mm). The wireless **gigabyte (WiGig) standard (IEEE 802.11ad)** for **millimetre-**wave wireless communication and vehicle radar **uses** 57 to 81 GHz. The WiGig **standard** has the highest data communication speed of 7 **Gbits** per second **over**

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short communication distances of approximately 10 m **together** with advanced properties such as security and power control. Presently, **the** microwave component industry uses several substrate materials compris**ed of both** hard and soft substrates [1-5]. Hard substrates **are** usually α-alumina, magnesia, aluminum nitride, fused silica (quartz), cordierite, GaAs **or** beryllia. The**se** ceramic (or quartz) substrates have the advantage of withstanding the localized heat **generated** during **the** wire-bonding process. Although hard substrates are highly isotropic, **exhibit high thermal conductivity (*k*) and comparable CTE and can withstand** very high operating temperatures, they **suffer from** brittleness, difficulty in machining and **the** relatively high cost of **the** chemically and thermally compatible conducting layer. Certain applications, such as patch antennas **and** base station circuitry, **require** substrates with larger sizes **and consequently** hard substrates are seldom used. In order to overcome these disadvantages, soft substrates based on ceramic filled thermoset plastic and thermoplastic have been developed [6,7]. These materials generally **have good** machinability, better shock resistance, low cost conduction/metallization layers, tailor-made properties and tight tolerances in their electrical properties. **Furthermore,** soft substrates based on some thermoplastic composites have more flexibility compared to thermoset based substrates. **These** are generally known as flexible substrates or

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laminates. The size, shape, interfacial properties, percolation level and porosity **of the filler particles have a crucial influence** on the end properties of the composites [6,7]. Thermoplastic materials have **a** melt temperature associated with them, whereas **the** dimensional behavior of thermosets **is** characterized by **their** glass transition temperature (Tg). **Generally,** thermoset matrix composites are preferred for low end applications whereas thermoplastic composites are the choice of preference for high end microwave circuit applications. The main advantages of thermoplastic matrix composites compared **to** thermoset matrix composites are **an** unlimited shelf life, reprocessability, chemical inertness, lower moisture absorption, higher service temperature, high environmental tolerance **and they do not require any curing** [6,7]. However, the processing difficulties and relatively high melt viscosity are important **disadvantages** of thermoplastic materials. The recent explosive growth in **the** wireless market and advances in high speed digital technology have **introduced** new thermoset **and** thermoplastic materials with superior electrical performance to address these needs. More recently, butyl and silicone rubber filled with low loss ceramic have been reported for flexible electronic applications [8-11]. The ceramic based hard substrates and polymer or rubber filled with ceramic materials have suitable relative permittivity and loss tangent at **the** lower end of the microwave frequency regime [5].

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However, as the frequency increases the loss tangent also increases although the relative permittivity is not much affected [2,5,6]. **Consequently,** these materials are unsuitable for **millimetre** wave or high frequency applications **and** there is an urgent need **to find** suitable polymer-ceramic composites with low loss **tangents in the** mi**llimetre** wave frequency regime. Silicon is reported as one of the most transparent materials for electromagnetic f**ields** at high frequencies [12-14]. **Float** zone as-grown silicon has a relative permittivity of 11.6 and **a** loss tangent of about 10^{-4} (quality factor (Q) of 17800 at 49 GHz) [15]. The present paper reports the preparation, characterization and high frequency dielectric properties of polypropylene filled with silicon powder. Polypropylene is selected as the polymer matrix **as it has a** low loss tangent (10^{-4}) with **a** relative permittivity of about 2.2 in the microwave/mm wave frequency region.

2. Materials and methods

In the present work, silicon **from wafers grown by the float zone method was** used as the particulate filler. The silicon wafers were crushed and powdered by ball milling using zirconia balls for about 12 hrs. The resultant silicon powder **had a wide** particle size distribution ranging from 10 to 100 μm. Polypropylene (PP) powder and silicon filler were used as the starting materials for the preparation of **the** PP/ Silicon

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composites. **C**ommercial grade PP granules supplied by Reliance **I**ndustries **L**imited, India **were** used as the starting material. Fine PP powders were prepared by dissolving the PP granules in Xylene (95%, Merck) at a temperature of 130°C **with** continuous stirring **from** a magnetic stirrer. The dissolved **material was** taken out, dried, mixed with **the** Si powder and **rigorously** ground. The PP/Si composites were fabricated **by** compression molding using a hydraulic laminating press **and** a 125 mm x125 mm size stainless steel die set. The optimized pressure and temperature employed for hot pressing were 90 kg/cm^2 and 180°C for 20 **minutes,** respectively. **P**lanar composite **125x125x1 mm** laminates were **obtained. The surface** morphology and filler distribution in the composite samples were studied using a scanning electron microscope (Carl Zeiss, EVO 18 Research, UK). The low frequency measurements were made without any metalization, employing **an** Agilent Technologies 16451B Dielectric Test Fixture connected to **a** HP4294A **precision impedance analyzer.** The dielectric properties were measured in the microwave frequency range up to 35 GHz using different split Post Dielectric Resonators (SPDR) connected to **an** Agilent Technologies PNA-X vector network analyzer. The coefficient of thermal expansion of the samples **was** measured in the temperature range 30-120^{o}C using an EXSTAR 6000 model thermo mechanical analyzer (SII Nano technology INC., Japan). The thermal

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diffusivity and heat capacity were measured by laser flash analysis (LFA 427, Netzsch, Germany). The thermal **conductivity (**TC) was calculated using the data of thermal diffusivity, bulk density and heat capacity using the relation TC=α x ρ x Cp where α is the thermal diffusivity **and** ρ and Cp are **the bulk density and** specific heat capacity of the composites, **respectively**. The samples used had dimensions 10x10 mm^2 and thickness 1 mm and were carbon coated on both sides to enhance **the absorption of** laser flash light.

3. Results and Discussion

An SEM picture of **a** silicon wafer crushed using a mortar and pestle is shown in Fig. 1. Ceramic particulates with **a wide range of** particle sizes from 10 to 130 μm are observed in the SEM picture.

Fig. 1 SEM picture of powdered silicon **from** wafer

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Silicon powders were incorporated in the PP matrix **by** fine mixing and compression molding techniques. 20, 25, 30, 35 and 40 vol % **samples** of PP/Silicon composites were prepared in the 125 mm x125mm stainless steel die. SEM pictures of **the** compression molded PP/Silicon samples are shown in Fig. 2.

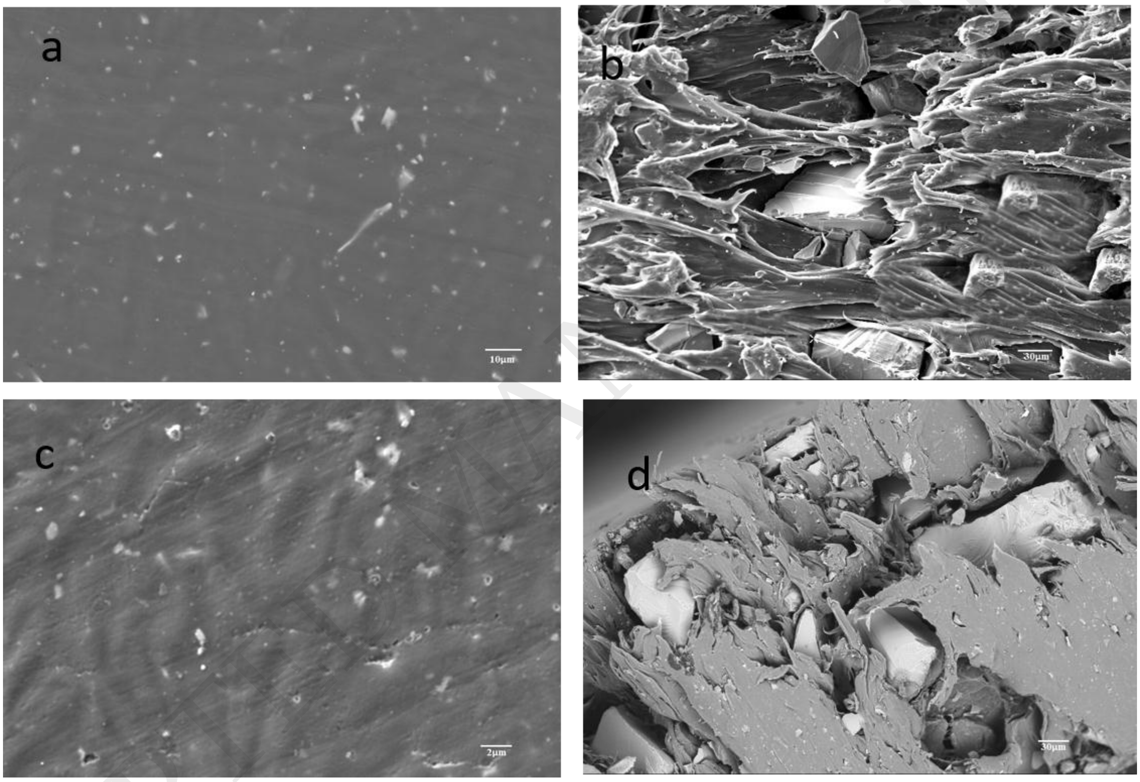

Fig. 2 (a) Planar and (b) cross sectional (fractured) micrographs of 30-vol % silicon filled PP **composite** (c) Planar and d) cross sectional (fractured) micrograph of 40 vol% silicon filled **PP composite**

It is evident from the planar microstructure (Fig 2(a)) of the 30 vol % silicon filled PP composite that the ceramic particulates **were** uniformly distributed in the polymer

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matrix with a nearly **pore-free** microstructure. The cross sectional **pictures also show a** fine distribution of Si particles with varying sizes. As the volume fraction of Si increas**ed** in the PP matrix, porosity **started to appear** in the composite. **This** is clearly evident from the planar microscopic image of the 40 vol % Si filled PP **composite** (Fig. 2(c)).

Low frequency measurements **were** performed using an impedance analyser and capacitive dielectric test fixture. In the capacitive measurements the sample under test **was** inserted between the two electrodes of an air capacitor and, together with the air gap above, the sample **created** a double-layer capacitor. **A schematic** of **the** capacitive measurement cell used in the experiments is show in Fig.3.

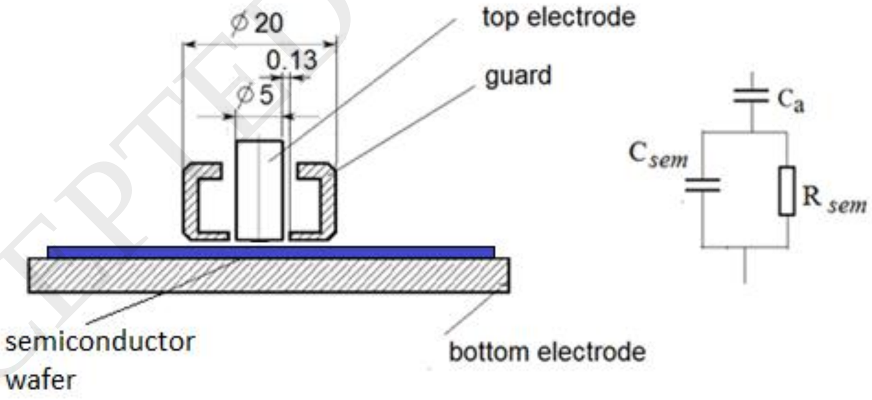

Fig.3. Agilent Technologies 16451B Dielectric Test Fixture with **5 mm diameter** guarded electrode **that was** used in the **experiments**.

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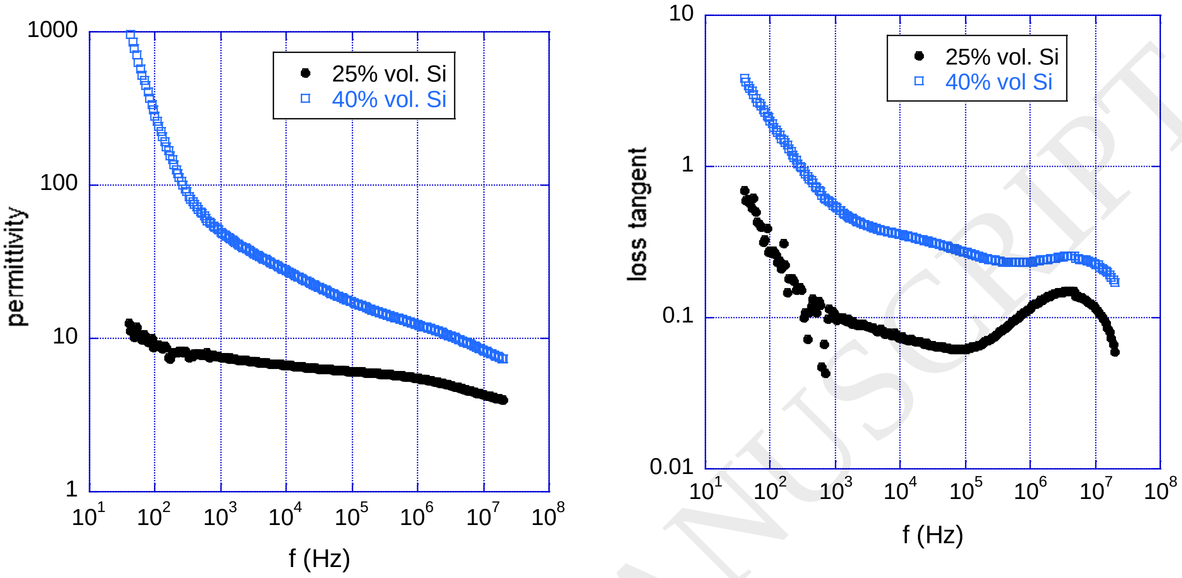

a) b)

Fig.4. Variation of (a) relative permittivity and (b) loss tangent as a function of frequency up to 15 MHz **(low frequency measurements).**

The dielectric properties of two representative samples 25 and 40 vol% are shown in Fig. 4. **The relative** permittivity of the composites sh**owed** a decreasing trend with respect to frequency up to 15 MHz. The change in relative permittivity **was** more for the 40 vol% composite as compared to 25 vol% silicon filled samples. This could be due to the space charge contribution from the **greater** amount of interface region in the 40 vol% filled samples. This was further evident from the frequency versus loss tangent graph.

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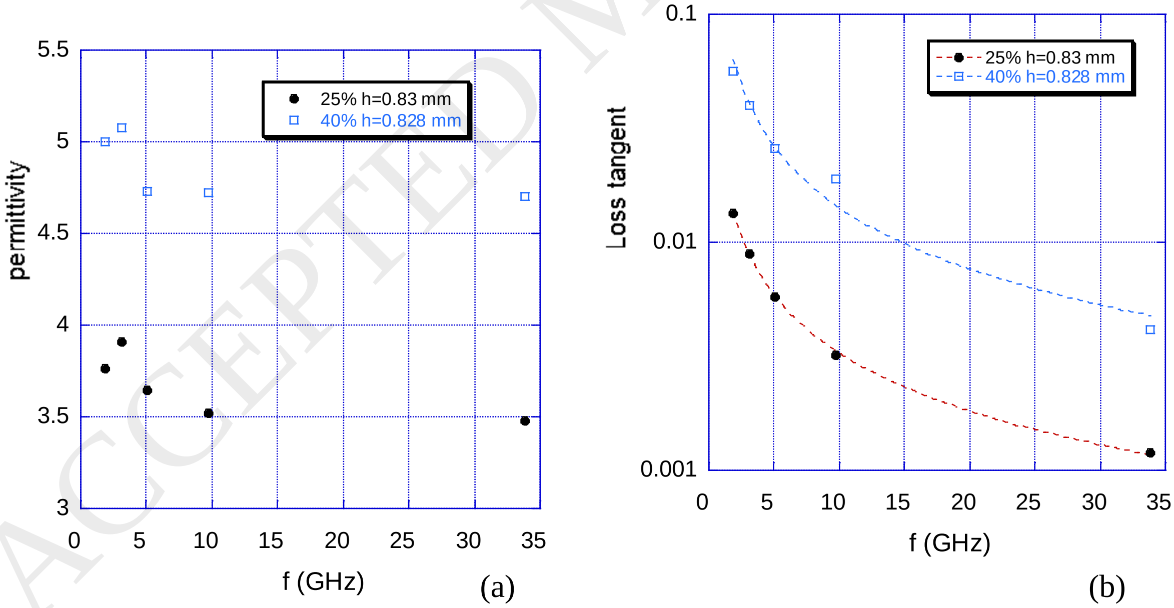
As the frequency increase**d**, the dispersion in the relative permittivity also **showed** a decreasing trend. The loss tangent also **showed a** linear decrease with respect to frequency with a relaxation peak around 10^6-10^7.

The frequency dependence of relative permittivity and loss tangent measured at high frequencies on two samples is shown in Fig. 5. These experiments **were** performed using five SPDR's having nominal frequencies of 1.9 GHz, 3.1 GHz, 5 GHz, 10 GHz and 35 GHz. It **was** seen that the loss tangent **was governed** by conductor losses in the silicon particles. It is expected that at higher frequencies losses will be much lower.



(a) (b)

Fig.5. (a) Relative permittivity and (b) dielectric loss tangent of PP-Si samples at frequencies up to 35 GHz

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The relative permittivity of the composite samples **remained more or less constant** from 10 GHz to 35 GHz (Fig. 5). Interestingly, the loss tangent of the composites e**xhibited** a sharp decrease with respect to **an** increase in frequency. It is inferred that such a variation is mainly due to the relatively lower loss tangent of the Si particulates at higher frequencies. The as-grown float zone silicon **had** a resistivity of about 80 kΩ.cm. The loss tangent of a material includes losses due to intrinsic and extrinsic factors and it is also affected by the conductivity of the material (conductor loss). The conductor loss decreases as the frequency increases [16]. It is well established that the loss tangent in silicon decreases with increase in frequency [14-18] due to **a reduction of** the conductor losses. Fig. 6 shows the variation of resonant frequency shift and loss tangent of PP-25% Si **composite as a function of temperature in the range 23-123 ^{o}C** measured using an SPDR operating at 4.8 GHz. It gives the temperature coefficient of permittivity to be about -21 ppm/^{o}C. It should be noted that the relative resonance frequency shift variations of the SPDR shown in Fig.6a **were** similar to the relative permittivity variations of the sample. This is quite different **from** the case of measurements of bulk samples acting as dielectric resonators. The loss tangent of the composite increases with increase in temperature and was due to the increase of the effective conductivity inside the Si particles.

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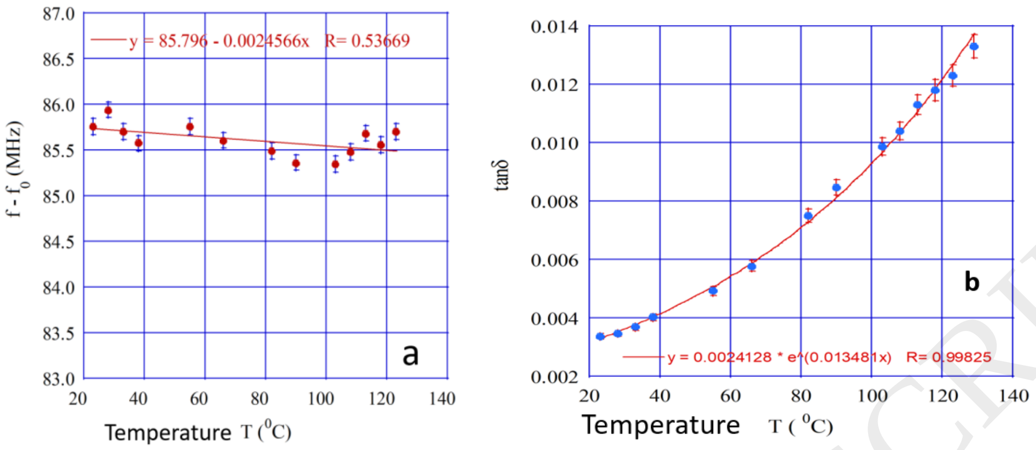

(a) (b)

Fig. 6. Variation of (a) resonant frequency shift and (b) loss tangent of PP-25%Si composite measured as a function of temperature using **an** SPDR operating at 4.8 GHz.

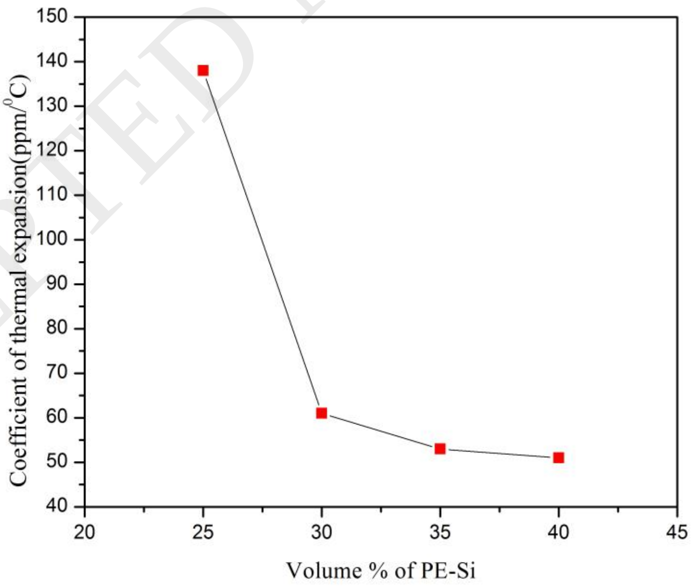

Fig. 7. Coefficient of thermal expansion of PP-Si samples **with varying vol% Si**

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Figure 7 shows the variation of CTE of the composite samples as a function of the volume fraction of Si loading. **A** decrease in CTE **was** noticed as a result of filler loading in the PP matrix. **A** CTE of 51 ppm/^{o}C **was** obtained for 40 vol% Si filled PP composites.

The thermal conductivity is also **an important parameter** for microelectronic applications. The heat transfer in a material is related to thermal conductivity, diffusivity and specific heat capacity. A low thermal conductivity value is generally preferred for minimizing heat losses in applications but a high thermal conductivity is required **to dissipate heat generated in a device or** when heat **needs to** transfer from one site to another. In the PP-Si composites, the thermal diffusivity slightly decreased with increase in temperature as shown in Fig. 8. The decrease **was** significant for the more **heavily** Si loaded samples **because Si** is a metalloid. The **increase in** specific heat of the **composites** with increase in temperature is shown in Fig. 9.

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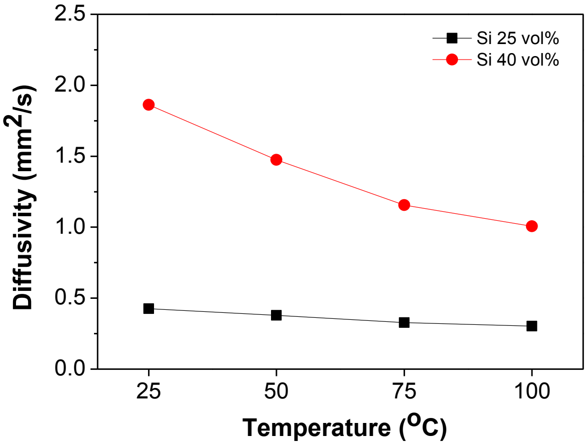

Fig.8. Variation of thermal diffusivity as a function of temperature

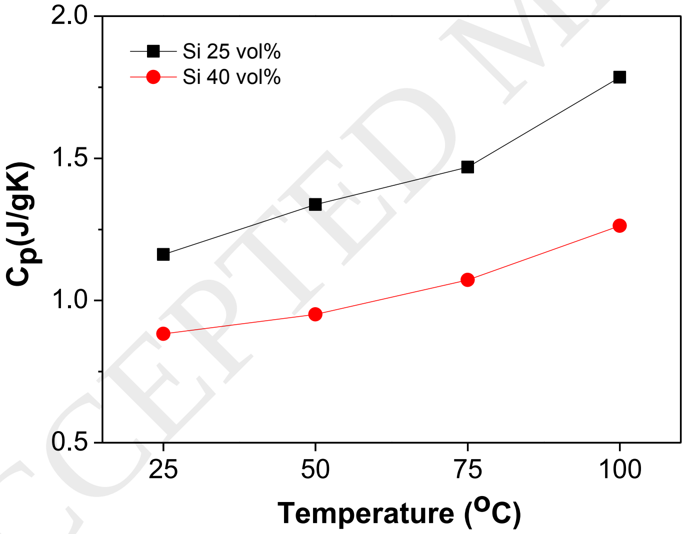

Fig. 9 Variation of specific heat as a function of temperature

Figure 10. shows the variation of thermal conductivity of the composites as a function of temperature. At room temperature (25°C), the PP-25 vol% Si composite h**ad**

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a thermal conductivity of 0.58 W/mK and the PP-40 vol% Si had 2.23 W/mK. The thermal conductivity **remained** nearly constant for the pp-25 vol% Si composite but it decreased significantly for the PP-40 vol% Si composite with an increase in temperature up to 100°C. The PP-25 vol% Si **had** an experimental density of 1.152 g/cm^3 and that of PP-40 vol% Si **was** 1.358 g/cm^3**.** **In general,** PP has a low thermal conductivity of about 0.22 W/mK whereas the Si has a high thermal conductivity of about 150 W/mK. The thermal conductivity increased to only about 0.58 W/mK for the composite with 25 vol% whereas it increased to 2.23 WmK when the Si content **was** increased to 40 vol%. The increase in thermal conductivity **was** relatively low considering the high thermal conductivity of Si. This is due to the fact that there is poor connectivity between the thermally conducting Si particles in the composite below the percolation threshold and the resultant properties depend on the PP matrix. However, it may be noted that the thermal conductivity of the PP composite increased by 163 % and 913 % for 25 and 40 vol% Si addition, respectively. It is clear from the present study that the thermal conductivity of pristine PP (0.22 (W/mK) can be considerably improved by the addition of silicon.

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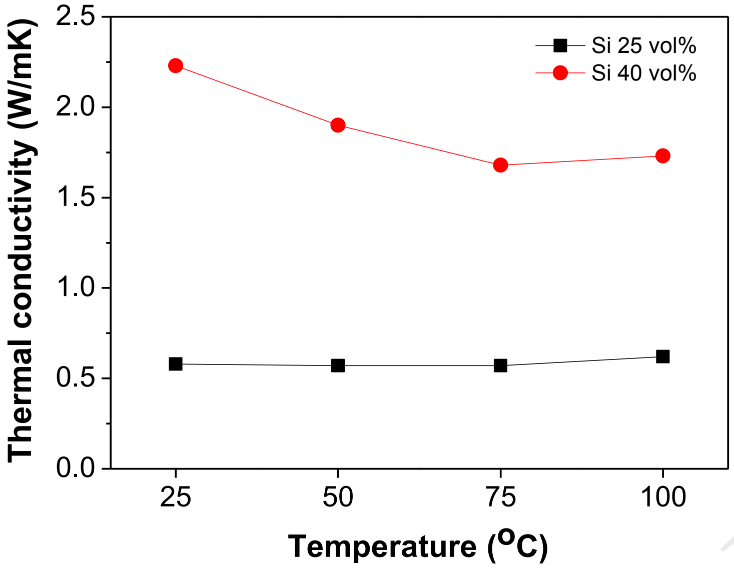

Fig. 10 Variation of thermal conductivity as a function of temperature

There are many factors, such as temperature, density, porosity, moisture, degree of crystallinity, orientation of grains, size of molecules **and** impurities, which can affect the thermal conductivity of a material [19,20]. The composite containing 40 vol% Si had higher porosity as evidenced by the SEM picture~~s~~ shown in Fig.2. It **has been** reported that the thermal conductivity (*k*) of polypropylene exhibits a smooth decrease with increase in temperature from 0.25 to 0.15 in the temperature range 25 to 100°C [19]. This behavior was attributed in terms of two mechanisms **with opposing** effects [19]: the specific heat increases slightly when the temperature rises, but the thermal diffusivity reduces due to the decrease in the phonon mean free path. The reduction in

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the polymer density (bulk density) with temperature also contributes to the reduction in thermal conductivity. The net effect of these mechanisms is a smooth decrease of the thermal conductivity with respect t**o a** rise in temperature. The thermal conductivity of any material is dependent on two things: (a) the motion of free electrons (b) lattice vibrations [19,20]. In metals, the thermal conductivity is mainly dependent on the motion of free electrons. The molecular vibrations increase with an increase in temperature and thus increase the mean free path of **the molecules, hence** obstructing the flow of free electrons. This **reduces** the thermal conductivity. The heat conductivity in non-metals is mainly due to phonons (lattice vibrations). Hence the thermal conductivity of non-metals is nearly a constant at low temperatures.

The PP-Si composite **had a** suitable relative permittivity, a low loss tangent which d**ecreased** with increasing frequency and a relatively high thermal conductivity. Such flexible composite laminates are ideal candidates for IOT and 5G applications. It has recently **been** reported that high energy proton irradiated Si can have a high resistivity and is highly transparent to electromagnetic waves at frequencies up to far infrared frequencies [12,13]. The increase in resistivity is due to the formation of both shallow and deep radiation defect centers that are responsible for the charge compensation in the material [21]. This high resistivity silicon has a high quality **factor** (low loss tangent) in

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the **millimetre** wave frequency range [14,15,17] and **the use of such** high resistivity Si may further improve the loss tangent **in the composites.**

4. Conclusion

Composites of polypropylene containing 25 and 40 vol % Silicon **were** fabricated by compression molding using a hydraulic laminating press. The CTE of the composites **decreased** with increasing amounts of silicon. The thermal conductivity was improved by the addition of silicon. The composite containing 40 vol% Si had a relative permittivity of 4.1, **a** loss tangent of 0.006 at 35 GHz, a CTE of 51 ppm/ºC and a thermal conductivity of 2.23 W/mK. The microwave dielectric properties and thermal properties **could** be tailored by changing the Si content in the PP matrix. The relative permittivity rem**ained** nearly constant but the loss tangent decreased with increasing frequency. These results show that the loss tangent of PP-Si composites **decreases** as a function frequency whereas other PP-composites reported in the literature show an **increase** in loss tangent with increasing frequency. This decreasing tendency of loss tangent with increase in frequency indicates the possibility of **the use of these polypropylene composites** in microwave and millimetre wave communication systems. **In addition,** they are important candidate materials for the Internet of Things (IOT) and 5G applications.

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Figure captions

Fig. 1 SEM picture of powdered silicon from wafer

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Fig.3. Agilent Technologies 16451B Dielectric Test Fixture with **5 mm diameter** guarded electrode that was used in the experiments

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Fig. 7. Coefficient of thermal expansion of PE-Si samples **with varying vol%Si**

Fig.8. Variation of thermal diffusivity as a function of temperature

Fig.9. Variation of specific heat as a function of temperature

Fig. 10. Variation of thermal conductivity as a function of temperature

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