THE CHEMISTRY OF CARE

Index Matching Silicone for High Brightness LED Packaging

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Although silicone technology has been around for over 5 decades, this unique material continues to find usage in new applications. Its various functionalities allow its usage as fuel resistant gaskets, inert implantable devices in the body, coatings for Atomic Oxygen protection in space, and for interocular lenses for cataract disease. This paper will examine various silicones as effective encapsulants and lenses for High Brightness LEDs (HB LEDs). Physical and optical characteristics will be tested to determine which materials may prove to be the best.

Polymer Chemistry

The term 'Silicone' is actually a misnomer. In organic Chemical nomenclature, the suffix '-one' delineates a molecule has a double bonded atom of oxygen to a carbon atom. Scientists initially believed that silicone materials contained double bonded oxygen to silicon atoms, hence the use of 'silicone.' Later, this structure was proven incorrect. While 'Silicone' is still used commonly, a more technically correct name is polysiloxane. The diagram below shows their typical structure:
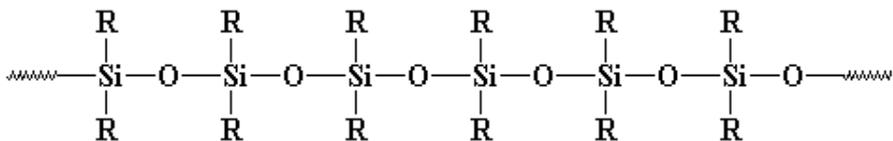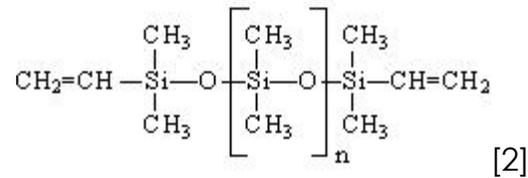

[1]

R=methyl, phenyl (aromatic carbon ring), 1,1,1-trifluoropropyl, ethyl

The variety of side groups allow polysiloxanes to be used in a wide array of applications, providing a variety of different properties. Different polysiloxanes can provide excellent properties that can be chosen according to the specific application; temperature stability (-115 to 260°C), fuel resistance, optical clarity (with refractive indexes as high as 1.60), low shrinkage (2-%), and low shear stress (2).

Different types of silicones, or polysiloxanes, and their property advantages include:

Dimethyl silicones, or dimethylpolysiloxanes, are the most common silicone polymers used industrially. These types of polymers are the most cost effective to produce and generally yield good physical properties in silicone elastomers and gels. The polymer pictured below contains vinyl endgroups that participate in a platinum catalyzed addition reaction (see section on *Cure Chemistry* for more information).
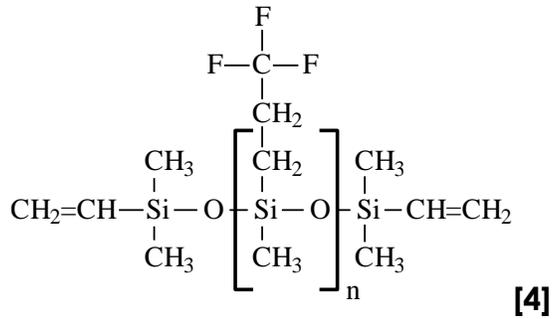
$$CH_2=CH-\underset{\underset{CH_3}{|}}{\overset{\overset{CH_3}{|}}{Si}}-O\left[\underset{\underset{CH_3}{|}}{\overset{\overset{CH_3}{|}}{Si}}-O\right]_n\underset{\underset{CH_3}{|}}{\overset{\overset{CH_3}{|}}{Si}}-CH=CH_2$$

[2]

For index matching purposes, all dimethylpolysiloxanes have a refractive index of 1.40, 25°C at 598nm.

Phenyl silicone systems contain diphenyldimethylpolysiloxane co-polymers. The phenyl functionality boosts the refractive index of the polymers from 1.40 upwards to 1.60. There are limitations, the steric hinderance of the large phenyl groups prohibit significantly high concentrations of diphenyl units on the polymer chain. Silicone polymers with diphenyl functionality with refractive index of 1.46 are useful in bio-photonic applications (e.g., intraocular lenses) in creating a thin lens. The diagram below shows a typical structure for a diphenyl dimethyl polysiloxane:

[3]
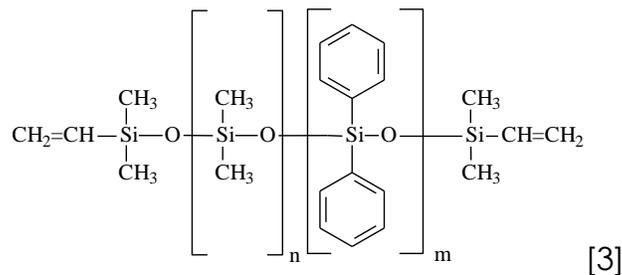
Fluorosilicones are based on trifluoropropyl methyl polysiloxane polymers and are used for applications that require fuel or hydrocarbon resistance. The trifluoropropyl group contributes a polarity to the polymer, resulting in swell resistance to gasoline and jet fuels. For photonic applications, the refractive index is 1.38 at 25°C at 598nm. While some fluorosilicones contain 100% trifluoropropylmethylpolysiloxane repeating units, other systems contain a copolymer of the fluorosiloxane units. Adjusting the amount of trifluoropropyl methyl siloxane units in the polymerization phase provides optimal performance in specific applications. The diagram below shows a typical structure for a 100%fluoropolysiloxane:

$$
\begin{array}{c}
\text{F} \\
| \\
\text{F}-\text{C}-\text{F} \\
| \\
\text{CH}_2 \\
\end{array}
$$

$$
\text{CH}_2{=}\text{CH}-\underset{\underset{\text{CH}_3}{|}}{\overset{\overset{\text{CH}_3}{|}}{\text{Si}}}-\text{O}\left[\underset{\underset{\text{CH}_3}{|}}{\overset{\overset{\text{CH}_2}{\overset{|}{\text{CH}_2}}}{\text{Si}}}-\text{O}\right]_n\underset{\underset{\text{CH}_3}{|}}{\overset{\overset{\text{CH}_3}{|}}{\text{Si}}}-\text{CH}{=}\text{CH}_2 \qquad \textbf{[4]}
$$

Material Composition

While the polymer chemistry and structure of polysiloxanes provide the different types of silicones outlined above, they also allow those different types of silicones to appear in a wide variety of material compositions. This broad range of material compositions makes silicone a viable option to endless numbers of photonic applications. Some silicone material compositions and their typical applications include:

Silicone Fluids are non-reactive and reactive silicone polymers formulated with dimethyl, methylphenyl, diphenyl, or trifluoropropylmethyl constituent groups, with refractive index ranging from 1.38 to 1.60. These materials' viscosity depends largely on molecular weight of the polymer and steric hinderance of functional groups on the polymer chain and can range from 100cP (a light oil) to an extremely thick gum polymer. Fluids are typically used to fill air gaps in high temperature lens assemblies in order to displace dust, permit cooling action or reduce interfacial reflections. Also to fill lenses or microchannel waveguides because of the large thermo-optic coefficient of 2 to 5 x 10-4/°C. Optical fluids can be used as the working medium in bubble-action optical switches or attenuators.

Silicone Thixotropic Gels are comprised of an optical fluid immobilized in a nanoparticle powder. These gels have no curing characteristics and by nature they are thixotropic and do not have well-defined viscosities. At rest they are mechanically stable and will not migrate. Due to the index matching limitations of the nanoparticle powder, these materials are available from 1.46 to 1.59 refractive indices only. Their primary use is for improving the return loss in a single mode mechanical fiber splice.

Silicone Curing Gels contain reactive silicone polymers and reactive silicone crosslinkers in a two-part system. When mixed together these materials are designed to have a very soft and compliant feel when cured and will stick to substrates without migrating. Viscosities can be

adjusted with the molecular weight of the polymers from 200 – 10,000cP. Depending on the functionality of the polymer, optical index matching can be formulated from 1.38-1.57. For HBLED applications this allows for the optimal light to come out of the die while protecting it from dust, moisture, vibration and changes in temperature. The yield strength of the gel is low enough to permit wire bonds to slice through during thermally induced micromotion without risking wire bond failure. Steps need to be taken to manufacture these materials with minimal outgassing and low ionic species. Other applications besides encapsulating HBLEDs include potting of packaged modules such as transponders, transceivers and detector arrays.

Silicone Thermosets fall into two categories, moldable elastomers and adhesives. Like the Gels, these two-part systems contain reactive polymers and crosslinkers that cure up to a rubbery type hardness. Most will cure at room temperature, however some need heat to cure. To impart increased physical properties, typically these materials have higher viscosities. The moldable materials can be casted or injection molded into HBLED lenses. They have inherently stronger physical properties than the gels and can work as excellent adhesives in optical applications. Special versions of these can be produced to have extremely low outgassing for electronic and aerospace applications. These also can have the broad refractive index range of 1.38 – 1.57.

Silicone resins, can range in structure from a highly branched amorphous resin to a regular ordered silsesquioxane, T Cube. Resin viscosities range from pourable to millions of centipoises. The empirical formula is:
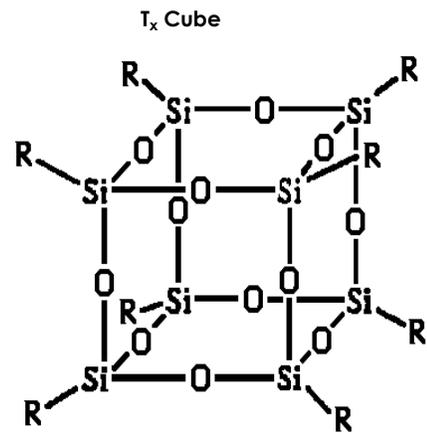
$$R - Si - O_{1.5} \qquad \text{[5]}$$

Both the Polysilsesquioxane and T-resin names can be derived from the empirical formula. The root "sesqui" indicates the one and a half stoichiometry of the oxygen bond to silicon. T-resin indicates the trisubstitution of silicon by oxygen. Silicone resins are also named by the organic, or "R," group.
Sample Resin Structures :
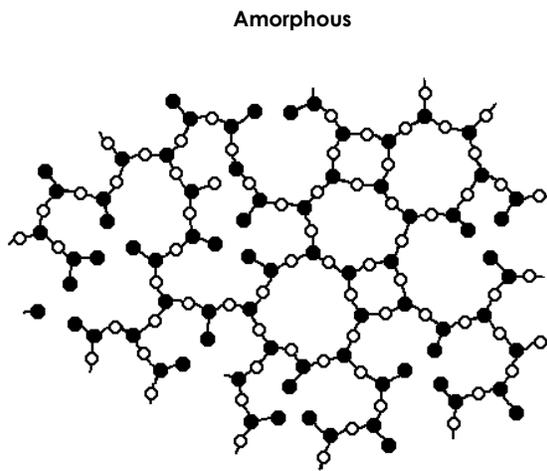
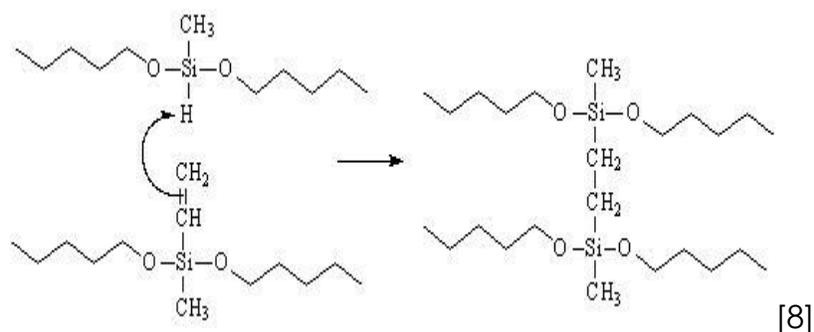|	
[6]	[7]

These materials when cured can give very hard durometers up to Type D. The phenyl content can be adjusted providing refractive index from 1.40-1.57.
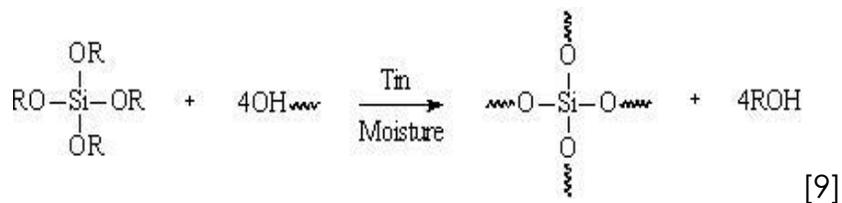
Cure Chemistry

When a manufacturer in the HB LED industry chooses a material for a specific application, material properties aren't the only deciding factor. That manufacturer also has to examine how the material is used. Inconvenience in production or material by-products can make a chosen material ineffective for a specific application. Silicones, however, can be designed around various cure chemistries to accommodate different production needs. Silicone systems can cure by platinum catalyzed addition cured systems, tin condensation cure systems, peroxide cure systems, or oxime cure systems. Some of the oldest cure chemistry used with silicones utilizes an acetoxy tin condensation cure system, such as used in household bathroom caulk. These systems yield a vinegar-like smell (acetic acid), a byproduct of the reaction. For various reasons as described below, platinum systems are the most appropriate for HBLED applications.

Platinum catalyzed silicones utilize a platinum complex to participate in a reaction between a hydride functional siloxane polymer and a vinyl functional siloxane polymer. The result is an ethyl bridge between the two polymers. The reaction mechanism is pictured below:

CH₃ structure [8]

$$CH_3 \quad O-Si-O \quad H \quad CH_2 \quad CH \quad O-Si-O \quad CH_3 \longrightarrow CH_3 \quad O-Si-O \quad CH_2 \quad CH_2 \quad O-Si-O \quad CH_3$$

[8]

Platinum systems are often cured quickly with heat, but can be formulated to cure at low temperatures or room temperature if necessary. The advantages of these systems include a flexible cure schedule and no volatile byproducts. The possibility of inhibiting the cure is the main disadvantage of platinum systems. Inhibition is defined as either temporarily or permanently preventing the system from curing. Some types of inhibitors are purposefully added to these systems to control the rate of cure. However, contact with tin, sulfur, and some amine containing compounds may permanently inhibit the cure. Compounds that inhibit the cure can be identified easily by attempting to cure a platinum catalyzed system in contact with the compound, as inhibition results in uncatalyzed regions of elastomer systems or inconsistency in cure over time.
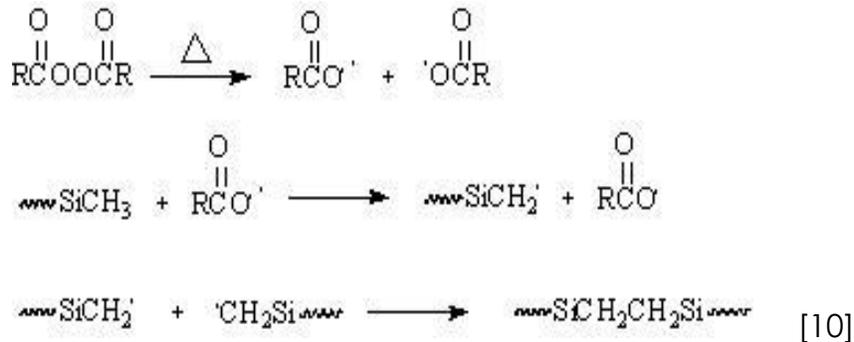
Tin condensation systems involve hydroxyl functional polymers and alkoxy-functional crosslinking compounds. The alkoxy functional crosslinker first undergoes a hydrolysis step and is left with a hydroxyl group. This hydroxyl group then participates in a condensation reaction with another hydroxyl group attached to the polymer. The reaction can proceed without the assistance of the tin catalyst, but the presence of the catalyst boosts the rate of reaction. The reaction mechanism is pictured below:

$$RO-\underset{OR}{\overset{OR}{Si}}-OR \quad + \quad 4OH\text{---} \quad \xrightarrow[\text{Moisture}]{\text{Tin}} \quad \text{---}O-\underset{O}{\overset{O}{Si}}-O\text{---} \quad + \quad 4ROH$$

[9]

The main disadvantages of condensation systems is the leaving group, shrinkage and long cure time, as several days are often required for to completely cure an elastomer.

Peroxide catalyzed systems, have a reaction mechanism that involves a peroxide catalyst and either methyl groups or vinyl functional groups. The

peroxide catalysts create free radical species of the methyl and vinyl that can then form covalent bonds. Pictured below is the reaction mechanism involving a peroxide catalysis of two methyl groups:

$$RCOOCR \xrightarrow{\triangle} RCO\cdot + \cdot OCR$$

$$\sim\!\!\sim SiCH_3 + RCO\cdot \longrightarrow \sim\!\!\sim SiCH_2\cdot + RCO$$

$$\sim\!\!\sim SiCH_2\cdot + \cdot CH_2Si\sim\!\!\sim \longrightarrow \sim\!\!\sim SiCH_2CH_2Si\sim\!\!\sim \qquad [10]$$

Disadvantages include a lengthy post-curing step at high temperatures in order to remove the reaction's byproducts. Other disadvantages include the possibility of the catalyst interacting with active agents.

Physical Properties

We will focus on two of the material types, Curing Gels and Thermosets, and describe various physical properties attainable:

Durometer, the hardness of the cured material tested as penetration, Type "OO", Type "A", and Type "D". Curing Gels, designed to be loosely crosslinking materials, will have either a penetration or a Type "OO" value. Penetration, the softest durometer, is tested on a penetrometer. The penetrometer allows a defined foot, or probe, to push into the cured sample at a defined force producing a measurement.

Referencing ASTM D 2240, Type "OO" durometer is measured by curing a sample at least 0.25" thick and placing it on a test stand with Type "OO" indenter. The indenter is forced down into the material at a constant force and a measurement is obtained. Thermosets will have durometers in the Type "A" and Type "D" range, which is harder than Type "OO". These are tested just like the "OO" except using a Type "A" or Type "D" indenter. As an example, a pencil eraser has a Type "A" durometer of 60 and a hardwood desktop a Type "D" durometer of 90. Cured gels and thermosets can be formulated to within ± 10% of pratically any hardness, from a penetration of 25mm to Type "D" of 30. The hardness for the HB LED packager depends on the application and what is being achieved. For wire bond stress and die protection, a soft Type "OO" 10, may be used. To eliminate the need for a lens and provide more durability, a harder Type "A" 40 may be used.

Lap Shear, following ASTM D1002, this test demonstrates the adhesive strength of a material to stainless steel or other substrates that one would want. Each substrate of choice is cut into a lap shear configuration, 1 inch wide by 4 inches long. Six strips of each substrate are prepared to make 3 test panels.   Panels are cleaned with isopropanol to remove dirt, grease or particulates. To improve adhesion a primer is sometimes added to one square inch area on one end of each lap panel as described above and let to sit for at least 30 minutes. A bond thickness target of 5ml(0.005in) is used for applying the Curing Gel or Thermoset to the primed area of the panels. The two panels are pressed together forming a sandwich (See Figure 2).   The sandwiched panels are then cured as required.

Because of their weak crosslinking, most cured gels are not able to be tested in this way. Current data shows 1.46 refractive index, silica filled thermoset materials to have the best adhesion to aluminum at over 500psi. NuSil technology is currently involved in more extensive adhesion studies, looking at higher refractive index materials and many of the common substrates used in HB LEDs; silver, gold, ceramic and polyphthalamide (PPA).

 Typical equipment used to test for lap shear value is an ISTRON Model 1011 with MTS data acquisition and 1000-lb load cell installed.

Figure 1. Instron 5500 Series Tabletop models for 450 lb to 11,250 lb capacities. New model updating the 1011.
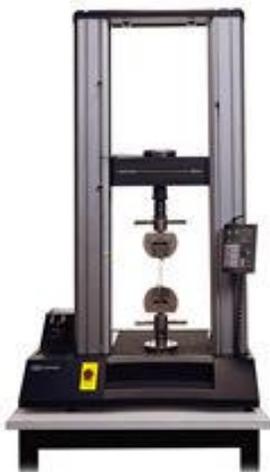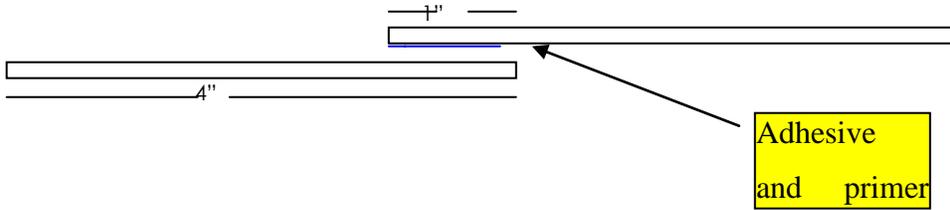

Figure 2. Lap joint.

1"

4"

Adhesive and   primer

Tear Strength, referencing ASTM D 624, a standard test to determine the tear properties of a cured material test sample to which a specific "cut" has been made to initiate the "tear". These figures typically given in pounds per inch(ppi), or their metric equivalent kN/m, describe the internal integrity of the material. Tear is a good descriptor of how well the material might wear under physical demands. Again, because of the low crosslinking, cured gels exhibit very low tear strength. The thermosets with refractive index up to 1.46 can reach 75ppi. Developmental materials have been produced with higher refractive indices, that exibit 30ppi. The limitation to higher refractive index materials is due to the inability to add reinforcing fillers.

Tensile Strength, based on ASTM D 412, a standard test to determine the strength of a material given a determined amount of strain. These figures typically corresponding to units of pounds per square inch or their metric equivalent mega Pascals, MPas. The higher the number the stronger the material. For Curing Gels, like tear, tensile values are too low to test. Also, similarly, 1.46 thermosets can get as high as 1200psi, while developmental materials >1.46 report 600psi.

Coefficient of Thermal Expansion, the measured expansion of a cured material under heat measured per 1°C rise or fall of temperature. Tested per ASTM D-3386-00, CTE reports above and below the glass transition temperature range as parts per million (PPM), i.e. LS-XXXX has a CTE value of 260 ppm per °C. Silicone materials typically have higher CTE values than other adhesive systems like epoxy, urethane, and acrylics, and in combination with their low modulus, allows them to be great bridge materials between two substrates with dissimilar CTE values. If a low CTE material were used as a bridge it would probably stress the substrates it is attached to and cause cracking.

Adhesive / Encapsulants	Coefficient of Thermal Expansion (x 10⁻⁶ mm/mm/°C)
Silicone	300
Filled Epoxy	70
PMMA	85

Unmodified Epoxy	70

Figure 3. Table of various CTE values for adhesives.

Volatile Content and Low Outgassing, based on ASTM D 2288 and E-595, determines the amount of material that does not get cured in a Cured Gel or Thermoset. This material does have the possibility of being outgassed under heat and pressure. Volatile content, ASTM D 2288, is determined by weighing a cured sample before and after being exposed to 150°C in a forced air oven for 3 hours.

   Low Outgas Testing, ASTM E-595, involves each material sample undergoing preconditioning, conducted at 50% relative humidity and ambient atmosphere for twenty-four hours. The sample is weighed and loaded into a compartment within a test stand (*Figure 4*). The sample is then heated to 125°C at less than 5×10^{-5} torr for 24 hours. Any volatile components of the sample outgas in these conditions. The volatiles escape through an exit port, and if condensable at 25°C, condense on a collector plate maintained at that temperature. The samples are post-conditioned in 50% relative humidity and ambient atmosphere for a twenty-four hour minimum.   The collector plate and samples are then weighed again to determine the percentage of weight change, determining Total Mass Loss(TML)% and Collected Volatile Condensable Material (CVCM)%. ASTM E-595 limit materials' (TML) to 1.0% and (CVCM) to 0.10%. To adhere to these requirements, NuSil Technology performs this
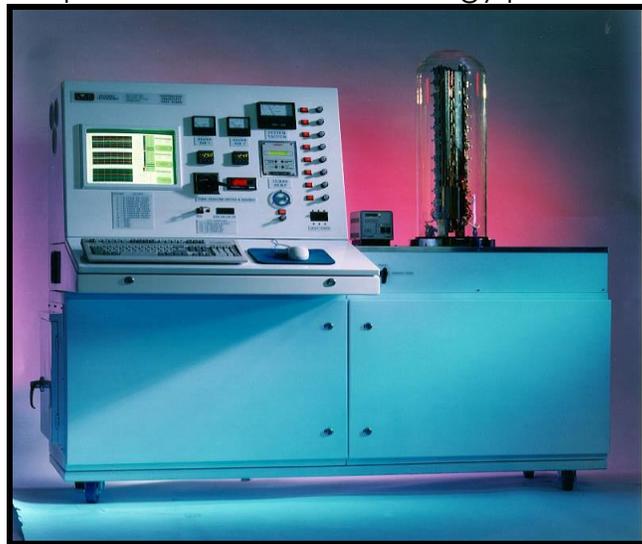
as a standard, lot-to-lot test.

Figure 4. Low Outgassing test stand.

Most cured silicone gels and thermosets can be formulated to have 0 volatile content and match the E-595 requirements for low outgassing. Most materials used in HBLEDs should have at least <0.5 volatiles.

Dielectric Strength, a measurement of the maximum voltage a cured material can withstand before electrical insulation breakdown or arc over occurs. Tested per ASTM D-149-97a, Dielectric Strength is reported as volts/mil, i.e. LS-XXXX has Dielectric Strength of 700 volts/mil. Materials that report a low dielectric strength are typically conductive. Most cured silicone gels and thermosets are in the 480-520 V/mil range.

Ionic Content, or Elemental Analysis of Trace Metals, is a water-extract test to determine the level of ionic contamination in a material. It commonly follows the requirements set out by MIL-STD-883E; <200ppm Chloride, < 50ppm Sodium, < 50ppm Potassium and
< 50ppm Fluoride. The test involves placing 3 grams of the cured material into a cleaned flask. The weight of the material is recorded to the nearest milligram and 150 grams of deionized water with low measured conductance is added. The flask is refluxed at 150ºC for 20 hours. Several of the silicone cured gels and thermosets have been tested and fall under the MIL-STD-883E requirements.

Thermal Gravimetric Analysis(TGA), is a method for determining the thermal degradation of a material by measuring the weight loss of a material as the material is heated. Results are plotted as material weight percent vs. temperature. Testing of several silicone cured gels and thermosets show TGA at 1% weightloss to be 300-360ºC.

Viscosity, a standard test to determine how thin or thick a liquid is. Used as a comparative figure, units are typically in centipoise, cps, or centistokes, cst, both essentially the same until you get into thicker liquids, or their metric equivalent milli Pascal seconds, mPas. Water is essentially 1cP, Pancake syrup 2500cP, honey 10,000cP, and peanut butter is around 250,000cP. For cured gels and thermosets, viscosity is measured prior to cure, often in the mixed form for a two-part system. Although there are limitations based on the overall properties, viscosity can be formulated to within ± 10% of any thickness desired. Current materials exist from 200 to 250,000cP.

Optical Properties

Refractive Index, the measurement of the speed of light traveling through a transparent material. It is measured at 589 nanometer(nm) wavelength(a.k.a. "the Sodium D line", or "nD") with a refractometer using the method of ASTM D-1218 at a fixed temperature of 25.0ºC. As previously mentioned, silicones have a refractive index range of 1.38-1.60.

Refractive Index of Common Substances

Material	Acronym	Tradenames	nD
Water			1.34
Polytetrafluoroethylene	PTFE	Teflon®	1.34
Magnesium Fluoride	MgF2		1.38
Fused quartz			1.46
Acrylate	PMMA	PLexiglass®	1.49
Cyclic Olefin	COC	Topas®	1.53
Diphenyldimethylpolysiloxanes	PVMQ		Up to 1.60
Polycarbonate	PC	Lexan®	1.59
Aluminum Oxide	Sapphire,ruby		1.76
Galium Nitride			2.5 to 2.7
AlGaInP			3.25

Figure 5. Refractive Index of common substrates

Refractive index versus change in temperature, is conducted at 25ºC to 50ºC in 5-degree steps. Typical data are reported as shown in Figure 6, and the least squares linear regression fit to the data for the thermo-optic coefficient (in units of dn/dT) is also calculated and provided on the chart.

Refractive Index vs. Temperature, 5 λ's
Lightspan Encapsulation Gel
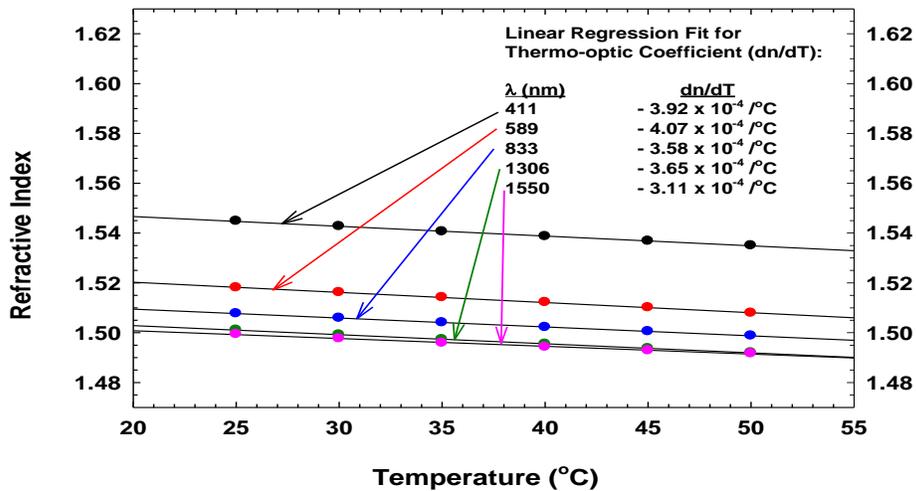*LS-3252, Lot# 20423-0417*

Figure 6

Refractive Index versus change in Wavelength, data are measured at 411nm, 589nm, 833nm, 1306nm and 1550nm at 25.0ºC and presented as

shown in Figure 3. Coefficients are also printed on the plot for a Sellmeier dispersion curve fit.
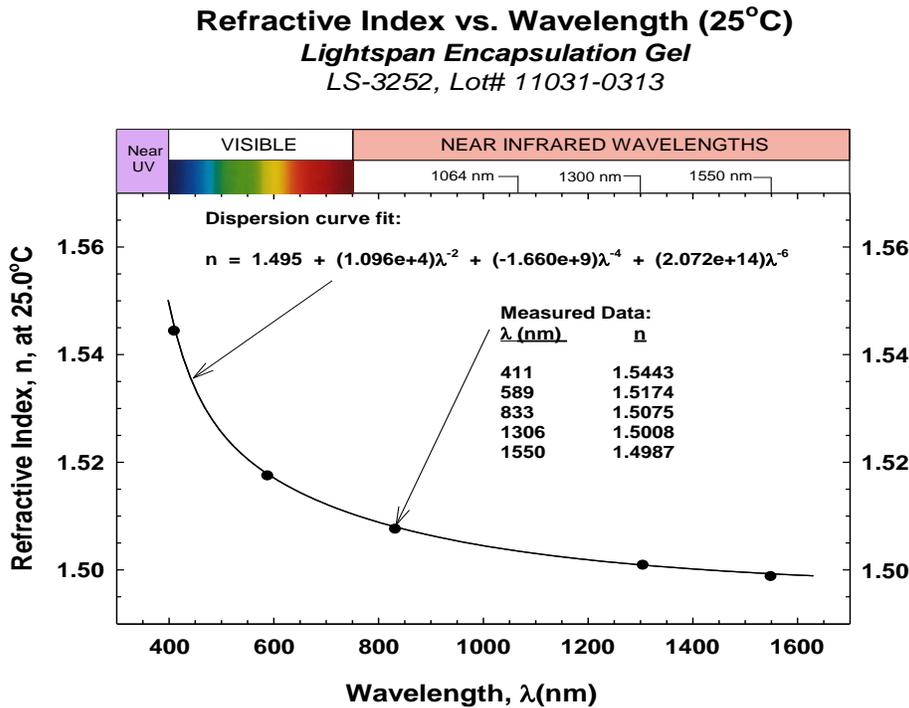
Refractive Index vs. Wavelength (25°C)
Lightspan Encapsulation Gel
LS-3252, Lot# 11031-0313

Near UV	VISIBLE	NEAR INFRARED WAVELENGTHS		
		1064 nm	1300 nm	1550 nm

Dispersion curve fit:

$$n = 1.495 + (1.096e+4)\lambda^{-2} + (-1.660e+9)\lambda^{-4} + (2.072e+14)\lambda^{-6}$$

Measured Data:

λ (nm)	n
411	1.5443
589	1.5174
833	1.5075
1306	1.5008
1550	1.4987

Refractive Index, n, at 25.0°C (y-axis: 1.50, 1.52, 1.54, 1.56)

Wavelength, λ(nm) (x-axis: 400, 600, 800, 1000, 1200, 1400, 1600)

Figure 7

Optical Absorption versus change in Wavelength, is measured across the wavelength range of 300nm to 1700nm, with 2nm resolution, using a spectrophotometer with the sample temperature at 25°C. Spectrophotometers come in many styles, though a typical spectrophotometer measures radiation transmission over a range of wavelengths starting in the ultra violet portion of the spectrum, passing through the visible, and extending into the infrared spectrum..   The discrete wavelengths of the spectrum used for the testing are generated by splitting a white beam of light with a diffraction grating (prism like device).   Rotation of the diffraction grating selects the portion of the spectrum illuminating the sample. In a double beam spectrophotometer the beam illuminating the sample is split into a reference beam and a sample beam. The reference beam is directed to a photodetector without passing through the sample. The reference beam is a measure of the incident radiation on the sample. The material being tested is placed in the path of the sample light beam, and only the light that passes through the material is detected at the photodetector. This is the transmitted radiation. Transmittance is defined as the incident radiation (Io) divided by the transmitted radiation (I):

$T = I/Io$,   and $\%T = 100 \times I/Io$
The Beer-Lambert Law defines Absorbance, A, as

A = log (I/Io) and
%A = 100 - %T

Absorbance is often more useful than transmittance since samples with low concentrations of absorbing species show a linear relationship between absorbance and the concentration of the absorbing species. Absorption is a function of distance so the path length of the sample is standardized to 1 cm. Absorption is correctly expressed as a certain per cent per unit length.

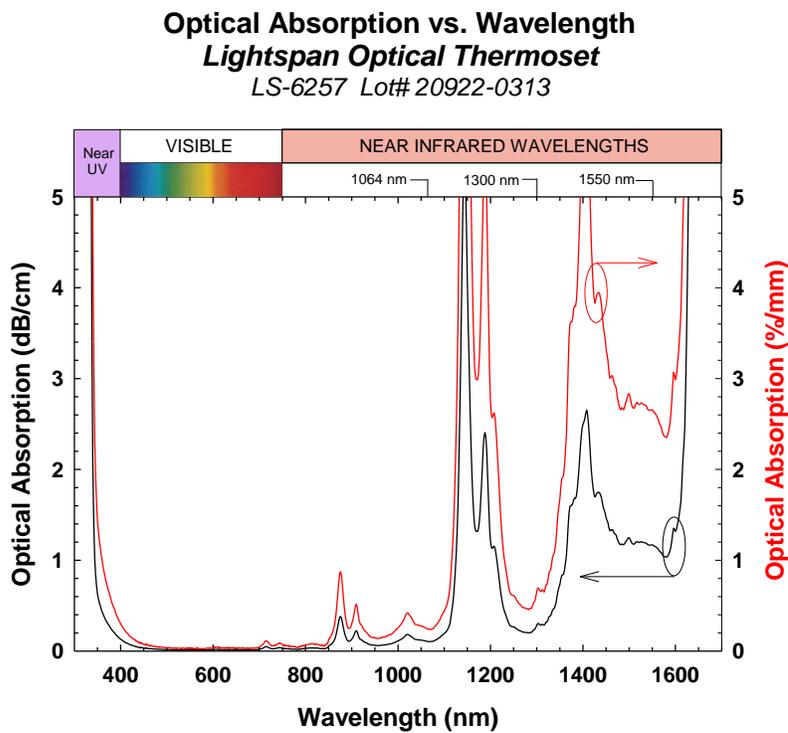An example of an absorption vs wavelength graph is shown in Figure 8.

Optical Absorption vs. Wavelength
Lightspan Optical Thermoset
LS-6257 Lot# 20922-0313

Figure 8

The graph shows that a high refractive index silicone thermoset has less than 1% absorption in the visible spectrum, the common spectrum for HB LEDs.

Conclusion

The theme of this paper demonstrates the many choices the HB LED package designer has with regards to silicone encapsulants and lens type materials. By modifying the polymer chemistry you can produce a

fluorosilicone with a refractive index of 1.38 or a methyl phenyl silicone with an index up to 1.60. Different forms of the same material can be produced, fluid, thixotropic gel, cured gel, thermoset and resin. Different cure systems can be designed, however for HB LEDs, the platinum systems offer the most advantages. Physical properties can also be custom formulated to specific requirements, adjusting the durometer, tear and tensile strength, viscosity and volatile content. The optical properties of these silicone materials can be characterized and favor use in HB LEDs. The chart in Figure 9(I would like to incorporate the Lightspan portion of our Engineering Selection Guide), gives a variety of current materials and their diverse properties. The best plan for a HB LED package designer is to involve the encapsulant or lens material provider early in the development process, so that the best material for the package can be found. After the fact it is often difficult to force fit a material into the package.

Future Studies

NuSil Technology is committed to remain in the forefront of HB LED packaging materials. We have ongoing tests with improving adhesion, UV effects on yellowing, both from sunlight and specific UV wavelengths, physical property improvements in the area of toughness with > 1.50 refractive index and finally a range of Type "D" hard materials for possible lens use.

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