US 20070031684A1

(19) United States
(12) Patent Application Publication
(10) Pub. No.: US 2007/0031684 A1
(43) Pub. Date: Feb. 8, 2007
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(54) THERMALLY CONDUCTIVE GREASE

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(21) Appl. No.: **11/195,953**

(22) Filed: **Aug. 3, 2005**

Publication Classification

(51) **Int. Cl.**
B32B 9/04 (2006.01)

(52) **U.S. Cl.** **428/447**; 428/450

(57) **ABSTRACT**

The invention relates to thermally conductive greases that may contain carrier oil(s), dispersant(s), and thermally conductive particles, wherein the thermally conductive particles are a mixture of at least three distributions of thermally conductive particles, each of the at least three distributions of thermally conductive particles having an average (D_{50}) particle size which differs from the other average particle sizes by at least a factor of 5

THERMALLY CONDUCTIVE GREASE

BACKGROUND

[0001] The invention relates to thermal interface materials and their use.

[0002] In the computer industry, there is a continual movement to higher computing power and speed. Microprocessors are being made with smaller and smaller feature sizes to increase calculation speeds. Consequently, power flux is increased and more heat is generated per unit area of the microprocessor. As the heat output of the microprocessors increases, heat or "thermal management" becomes more of a challenge.

[0003] One aspect of thermal management is known in the industry as a "thermal interface material" or "TIM" whereby such a material is placed between a heat source, such as a microprocessor, and a heat dissipation device to facilitate the heat transfer. Such TIMs may be in the form of a grease or a sheet-like material. These thermal interface materials also are used to eliminate any insulating air between the microprocessor and heat dissipation device.

[0004] TIMs typically are used to thermally connect a heat source to a heat spreader, that is, a thermally conductive plate larger than the heat source, in which case they are referred to as TIM Is. TIMs may also be employed between a heat spreader and a thermal dissipation device such as a cooling device or a finned heat sink in which case such TIMs are referred to as TIM IIs. TIMs may be present in one or both locations in a particular installation.

SUMMARY

[0005] In one embodiment, the invention provides a thermally conductive grease that comprises 0 to about 49.5 weight percent of carrier oil, about 0.5 to about 25 weight percent of at least one dispersant, and at least about 50 weight percent of thermally conductive particles. The thermally conductive particles comprise a mixture of at least three distributions of thermally conductive particles, each of the at least three distributions of thermally conductive particles having an average (D_{50}) particle size which differs from the other distributions by at least a factor of 5.

[0006] In another embodiment, the invention provides a method of making a thermally conductive grease of the invention that comprises the steps of providing carrier oil, dispersant, and thermally conductive particles, and then mixing the carrier oil (if present), dispersant, and thermally conductive particles together.

[0007] In one aspect, the carrier oil (if present) and dispersant are mixed together, and the thermally conductive particles are mixed sequentially, finest to largest average particle size into the carrier oil and dispersant mixture. In another aspect, the thermally conductive particles are mixed together, and then mixed into the carrier oil (if present) and dispersant mixture. In another aspect, a portion or all of the thermally conductive particles are pre-dispersed with a dispersant prior to mixing the thermally conductive particles into the carrier oil (if present) and dispersant mixture.

[0008] In another embodiment, the invention provides a microelectronic package comprising a substrate, at least one microelectronic heat source attached to the substrate, and a thermally conductive grease disclosed in this application on the at least one microelectronic heat source.

[0009] In one aspect, the invention provides the above microelectronic package further comprising a heat spreader and thermally conductive grease disclosed in this application between the microelectronic heat source and the heat spreader.

[0010] In another aspect, the invention provides a microelectronic package comprising a substrate, at least one microelectronic heat source attached to the substrate, a heat spreader, and a heat dissipation device attached to the heat spreader wherein a thermally conductive grease disclosed in this application is between the heat spreader and the heat dissipation device.

[0011] In another aspect, the invention provides a microelectronic package comprising a substrate, at least one microelectronic heat source attached to the substrate, a heat spreader, a thermally conductive grease disclosed in this application between the microelectronic heat source and the heat spreader and a heat dissipation device wherein thermally conductive grease is between the heat spreader and the heat dissipation device.

DETAILED DESCRIPTION

[0012] As used herein:

[0013] "Grease" means a material having a viscosity of greater than 1×10^4 cps (10 Pa.s) at 1/s shear rate and 20° C. and a viscosity of less than 108 cps at 1/sec shear rate and 125° C.

[0014] "Thermally conductive grease" means grease having a bulk conductivity of greater than 0.05 W/m-K as measured by the test method Bulk Thermal Conductivity described below.

[0015] All numbers are herein assumed to be modified by the term "about," unless stated otherwise. The recitation of numerical ranges by endpoints includes all numbers subsumed within that range (e.g., 1 to 5 includes 1, 1.5, 2, 2.75, 3, 3.80, 4, and 5).

[0016] Thermally conductive greases (TCGs) of the invention may contain one or more carrier oils. Carrier oil provides the base or matrix for the TCGs of the invention. Useful carrier oils may comprise synthetic oils or mineral oils, or a combination thereof and are typically flowable at ambient temperature. Specific examples of useful carrier oils include polyol esters, epoxides, silicone oils, and polyolefins or a combination thereof.

[0017] Commercially available carrier oils include HATCOL 1106, a polyol ester of dipentaerythritol and short chain fatty acids, and HATCOL 3371, a complexed polyol ester of trimethylol propane, adipic acid, caprylic acid, and capric acid (both available form Hatco Corporation, Fords, N.J.); and HELOXY 71 an aliphatic epoxy ester resin, available from Hexion Specialty Chemicals, Inc., Houston Tex.

[0018] Carrier oil may be present in the TCGs of the invention in an amount of from 0 to about 49.5 weight percent, and in other embodiments, from 0 to not more than about 20 or about 12 weight percent of the total composition. In other embodiments, carrier oil may be present in an amount of at least 2, 1, or 0.5 weight percent of the composition. Carrier oil may also be present in the TCGs of the invention in ranges including from about 0.5, 1, or 2 to about 12, 15, or 20 weight percent.

[0019] TCGs of the invention contain one or more dispersants. The dispersant(s) may be present in combination with carrier oil, or may be present in the absence of carrier oil. The dispersants improve the dispersion of the thermally conductive particles (described below) in the carrier oil if present. Useful dispersants may be characterized as polymeric or ionic in nature. Ionic dispersants may be anionic or cationic. In some embodiments, the dispersant may be nonionic. Combinations of dispersants may be used, such as, the combination of an ionic and a polymeric dispersant.

[0020] Examples of useful dispersants include, but not limited to, polyamines, sulfonates, modified polycaprolactones, organic phosphate esters, fatty acids, salts of fatty acids, polyethers, polyesters, and polyols, and inorganic dispersants such as surface-modified inorganic nanoparticles, or any combination thereof.

[0021] Commercially available dispersants include those having the tradenames SOLSPERSE 24000 and SOLSPERSE 39000 hyperdispersants, available from Noveon, Inc., a subsidiary of Lubrizol Corporation, Cleveland, Ohio; EFKA 4046, a modified polyurethane dispersant, available from Efka Additives BV, Heerenveen, The Netherlands; and RHODAFAC RE-610, an organic phosphate ester, available from Rhone-Poulenc, Plains Road, Granbury, N.J.

[0022] Dispersant is present in the TCGs of the invention in an amount of at least 0.5 and not more than 50 weight percent, and in other embodiments, not more than 25, 10, or 5 weight percent of the total composition. In another embodiment, dispersant may be present in an amount of at least 1 weight percent. Dispersant may also be present in the TCGs of the invention in ranges including from about 1 to about 5 weight percent.

[0023] TCGs of the invention contain thermally conductive particles. Useful thermally conductive particles include, but are not limited to, those made from or that comprise diamond, polycrystalline diamond, silicon carbide, alumina, boron nitride (hexagonal or cubic), boron carbide, silica, graphite, amorphous carbon, aluminum nitride, aluminum, silver, and combinations of any of them. Each of these particles are of a different type.

[0024] The thermally conductive particles used in the TCGs of the invention are a mixture of at least three distributions of thermally conductive particles. Each of the at least three distributions of thermally conductive particles have an average particle size which differs from the average particle size of the distribution above and/or below it by at least a factor of 5, and in other embodiments, at least a factor of 7.5, or at least a factor of 10, or greater than 10. For example, a mixture of thermally conductive particles may consist of: a smallest particle distribution having an average particle diameter (D_{50}) of 0.3 micrometers; a middle distribution having an average particle diameter (D_{50}) of 3.0 micrometers; and a largest distribution having an average particle diameter (D_{50}) of 30 micrometers. Another example may have average diameter particle distributions having average particle diameter (D_{50}) values of 0.03 micrometers, 0.3 micrometers, and 3 micrometers.

[0025] The thermally conductive particles used in the TCGs of the invention are a mixture of at least three distributions of thermally conductive particles resulting in at least a trimodal distribution. In such a trimodal distribution, the minima between the peaks (distance between the baseline of the peaks and the lowest point of the valley between distribution peaks) may be no more than 75, 50, 20, 10 or 5 percent of the interpolated value (height) between adjacent peaks. In some embodiments, the three size distributions are essentially non-overlapping "essentially non-overlapping" means that the lowest point of the valley is no more than 5% of the interpolated value between adjacent peaks. In other embodiments, the three distributions have only a minimal overlap. "Minimal overlap" means that the lowest point of the valley is no more than 20% of the interpolated value between adjacent peaks.

[0026] Typically, for a trimodal TCG, the average particle size for the smallest average diameter may range from about 0.02 to about 5.0 micrometers. Typically, the average particle size for the middle average diameter may range from about 0.10 to about 50.0 micrometers. Typically, the average particle size for the middle average diameter may range from about 0.5 to about 500 micrometers.

[0027] In some embodiments, it is desirable to provide a TCG having the maximum possible volume fraction thermally conductive particles that is consistent with the desirable physical properties of the resulting TCG, for example, that the TCG conform to the surfaces with which it is in contact and that the TCG be sufficiently flowable to allow easy application.

[0028] With this in mind, the conductive particle distributions may be selected in accordance with the following general principles. The distribution of smallest diameter particles should have diameters that are smaller than, or nearly bridge, the expected gap between the two substrates to be thermally connected. Indeed, the largest particles may bridge the smallest gap between substrates. When the particles of the largest diameter distribution are in contact with each other, a gap or void volume between the particles will remain. The mean diameter of the middle diameter distribution may be advantageously selected to just fit within the gap or void between the larger particles. The insertion of the middle diameter distribution will create a population of smaller gaps or voids between the particles of the largest diameter distribution and the particles of the middle diameter distribution the dimensions of which may be used to select the mean diameter of the smallest distribution. In a similar fashion, desirable mean particle dimensions may be selected for fourth, fifth, or higher order populations of particles if desired.

[0029] Each distribution of thermally conductive particles may comprise the same or different thermally conductive particles in each or any of the at least three distributions. Additionally, each distribution of thermally conductive particles may contain a mixture of different types of thermally conductive particles

[0030] The remaining voids may be thought of as being filled with carrier, dispersant(s) and other components with a slight excess to provide flowability. Further guidance in the selection of suitable particle distributions may be found in "Recursive Packing of Dense Particle Mixtures", Journal of Materials Science Letters, 21, (2002), pages 1249-1251. From the foregoing discussion, it will be seen that the mean diameters of the successive particle size distributions will preferably be quite distinct and well separated to ensure that they will fit within the interstices left by the previously packed particles without significantly disturbing the packing of the previously packed particles.

[0031] The thermally conductive particles may be present in the TCGs of the invention in an amount of at least 50 percent by weight. In other embodiments, thermally conductive particles may be present in amounts of at least 70, 75, 80, 85, 86, 87, 88, 89, 90, 91, 92, 93, 94, 95, 96, 97, or 98 weight percent. In other embodiments, thermally conductive particles may be present in the TCGs of the invention in an amount of not more than 99, 98, 97, 96, 95, 94, 93, 92, 91, 90, 89, 88, 87, 86, or 85 weight percent.

[0032] The TCGs and TCG compositions of the invention may also optionally include additives such as antiloading agents, leveling agents and solvents (to reduce application viscosity), for example, methylethyl ketone (MEK), methylisobutyl ketone, and esters such as butyl acetate.

[0033] The TCGs of the invention are generally made by blending dispersant and carrier oil together, and then blending the thermally conductive particles sequentially, finest to largest average particle size into the dispersant/carrier oil mixture. The thermally conductive particles may also be premixed with one another, and then added to the liquid components. Heat may be added to the mixture in order to reduce the overall viscosity and aid in reaching a uniformly dispersed mixture. In some embodiments, it may be desirable to first pretreat or pre-disperse a portion or all of the thermally conductive particles with dispersant prior to mixing the particles into the dispersant/carrier mixture.

[0034] The TCGs of the invention may be used in microelectronic packages and may be used to assist in the dissipation of heat from a heat source, for example, a microelectronic die or chip to a thermal dissipation device. Microelectronic packages may comprise at least one heat source, for example, a die mounted on a substrate or stacked die on a substrate, a thermally conductive grease of the invention on the heat source, and may include an additional thermal dissipation device in thermal and physical contact with the die, such as, for example, a thermal spreader. A thermal spreader may also be a heat source for any subsequent thermal dissipation device. The thermally conductive greases of the invention are useful to provide thermal contact between said die and thermal dissipation device. Additionally, TCGs of the invention may also be used in thermal and physical contact between a thermal dissipation device and a cooling device. In another embodiment, the TCGs of the invention may be used between a heat generating device and a cooling device, that is, without using a heat or thermal spreader in between. TCGs of the invention are useful in TIM I and TIM II applications.

EXAMPLES

Bulk Thermal Conductivity

[0035] Bulk thermal conductivity was measured generally in accord with ASTM D-5470-01 on the TCG samples using a Heat Transfer Tester, available from Custom Automation, Inc., Blaine, Minn. The Heat Transfer Tester was built according to Proposal Number 3M-102204-01 and included such features as: a vision system capable of measuring parallelism and gap between copper meter bars for up to 0.010 inch (0.254 mm) gaps, copper meter bars with 5 resistance temperature detector (RTD) sensors on each meter bar, a cooler to cool the cooled clamping block (to hold the cooled meter bar) having an operating range of from −20 to 100° C. and can hold the coolant temperature to ±0.02° C., a 25 IbF load cell mounted on a X-Y micrometer adjust positioning stage, a cooled clamping block (to hold the cooled meter bar) mounted on the load cell, a heated clamping block (to hold the heated meter bar) using resistive heating and has its temperature controlled by a controller and thermocouple, the ability to add weights above the heated clamping block to adjust the contact force on the meter bars from 5 to 50 N, and means to measure and record temperature, meter bar gap, and contact force at time intervals to a spreadsheet.

[0036] The vision system used to measure meter bar gap was calibrated as outlined in the operating procedures provided. The cooler was charged with a 50/50 blend of water and ethylene glycol. The gap between the copper meter bars was set at about 550 micrometers at room temperature. The heater set point was put at 120° C. and the cooler set point at −5° C., and the unit was allowed to equilibrate. The meter bar gap after equilibration was about 400 micrometers. The surfaces of the hot and cold meter bars were planarized using the individual meter bar tumbuckles until the gap between the meter bars read by each of the three individual cameras fell within a ±3 μm range.

[0037] An excess of each TCG sample tested was placed on the hot meter bar surface and smoothed across the entire face. The head was then closed and clamped into place, causing excess TCG sample to ooze out of the meter bar gap. This excess was removed with a paper towel or a fine cloth and the pins of the meter bars were cleaned to facilitate accurate measurement of the gap by the three vision cameras. The instrument was allowed to equilibrate for about 10 minutes as data was continuously recorded. The meter bar gap was lowered about 100 μm and excess TCG sample oozed out of the gap and was cleaned. The instrument was again allowed to equilibrate for about 10 minutes as data was continuously recorded. This sequence of lowering the meter bar gap in about 100 μm increments, cleaning, and recording data was repeated until a final reading was taken, typically at a meter bar gap of <100 μm. The meter bars were opened back up to about a 400 μm gap, cleaned, and the procedure was repeated for the next sample.

[0038] The data were recorded every 7-8 seconds by the instrument and contained a time/date stamp, the sample name, the force exerted on the TCG in the meter bar gap, each of the individual meter bar gap readings, and each of the 10 RTD sensor temperature readings. The file was downloaded into a spreadsheet for analysis. In the analysis, the last 10 data points recorded at the given gap were averaged, and these averages were used for the calculations.

[0039] The power flowing through the TCG sample was calculated using the known bulk conductivity of copper, the dimensions of the copper bars, and the locations of the RTD temperature sensors. Typically, the calculations indicated slightly different wattage flowing down the hot meter bar than down the cold meter bar; these two values were averaged for calculations extending to the TCG sample. The temperature at the surface of each of the meter bars was also extrapolated from a plot of the temperatures and the RTD sensor locations.

[0040] The power, the average of the three individual meter bar gaps, the temperature drop across the meter bar gap, and the cross sectional area of the hot/cold meter bars were then used to calculate the temperature gradient, the power flux, and then the thermal impedance for the TCG sample under those conditions.

[0041] These calculations were completed for each of the meter bar gaps at which the TCG sample had been tested, and the resulting thermal impedance and average gap data was plotted. A line was fitted to the data using spreadsheet software, and the bulk conductivity was calculated as the inverse of the lines' slope. The y-axis intercept and the slope were then used to calculate the thermal impedance at a 100 μm meter bar gap.

Viscosity

[0042] The viscosity data on selected samples was generated on a Rheometrics RDA3 viscometer (TA Instruments, Newcastle, Del.). The viscometer was run with disposable 1 inch (25.4 mm) diameter parallel plates in the log sweep mode starting at 0.5/sec initial shear rate, taking 5 points/decade up to 1000/sec shear rate. The gap was set at 0.5 mm for a run, and then lowered to 0.25 mm for a second run on some samples; on other samples the gap was set and run only at 0.25 mm. Temperatures of the runs were controlled to either 125° C. or 25° C. as indicated in the table below. Viscosities were recorded in mPa.s at a 1.25/sec shear rate.

Milling Procedure

[0043] Roughly 40 cc of 0.5 mm diameter yttria-stabilized zirconia beads (available from Tosoh, Hudson, Ohio or from Toray Ceramics, George Missbach & Co., Atlanta, Ga.) were put into the basket of a Hockmeyer HM-1/16 Micro Mill ("Hockmeyer mill") (Hockmeyer Equipment Corp., Harrison, N.J.). The desired MEK and dispersant (SOLSPERSE) were added to the mill chamber and stirred with an air mixer for at least 4 minutes so as to dissolve the dispersant in the solvent. The diamond particles were weighed into the chamber and the contents were stirred for an additional minute to wet out the diamond particles. The resulting mixture was then milled at the maximum speed of the Hockmeyer that avoided splashing. The resulting slurry was poured into a polyethylene container and the solvent was allowed to evaporate until it could not be detected by odor. Details of the compositions milled are shown below.

Diamond Particle Size	Mill	Mill Charges		
(D_{50}) (micrometer)	Time (min)	Methyl Ethyl Ketone (g)	SOLSPERSE 24000 (g)	Diamond Particles (g)
0.25	20	280	54	900
0.50	15	280	27	900
1.00	10	255	16.5	1100

[0044]

Glossary

Name	Description	Source
BYK 361	Polyacrylate copolymer leveling agent	BYK-Chemie USA, Wallingford, CT
2,2' Bypyridyl-ethylene bis-salicylimine	A chelating agent	Alfa Aesar, Ward Hill, MA
DP 1	Diamond particles having a D_{50} of 0.25 μm and a D_{50} of 0.50 μm	Tomei Diamond, Englewood Cliffs, NJ
DP 2	Diamond particles having a D_{50} of other than 0.25 or 0.50 μm	National Diamond Research Company, Chesterfield, MI
Ethylene bis-salycimine	A chelating agent	Strem Chemicals, Newburyport, MA
F180 SiC	Silicon carbide particles having a D_{50} particle size of 80 μm	Washington Mills Electro Mineral Corp., Niagara Falls, NY
GAFAC RE 610 (now RHODAFAC RE-610)	An ionic dispersant	Rhone-Poulenc, Granbury, NJ
GC 20000	Silicon carbide particles having a D_{50} of 0.3 μm	Fujimi Corporation, Nagoya, JP
GC 8000	Silicon carbide particles having a D_{50} of 1.0 μm	Fujimi Corporation
GC 4000	Silicon carbide particles having a D_{50} of 3.0 μm	Fujimi Corporation
GC 2000	Silicon carbide particles having a D_{50} of 9 μm	Fujimi Corporation
GC 1200	Silicon carbide particles having a D_{50} of 13.5 μm	Fujimi Corporation
GC 700	Silicon carbide particles having a D_{50} of 18 μm	Fujimi Corporation
GC 400	Silicon carbide particles having a D_{50} of 35 μm	Fujimi Corporation
HATCOL 1106	A polyol ester of dipentaerythritol and short chain fatty acids (carrier oil)	Hatcol Corporation, Fords, NJ
HATCOL 2300	A complexed polyol ester or pentaerythritols and short chain fatty acids (carrier oil)	Hatcol Corporation
HATCOL 2930	A diester of trimellitic anhydride and isodecyl alcohol (carrier oil)	Hatcol Corporation
HATCOL 2949	A diester of dimer acid and 2-ethyhexyl alcohol (carrier oil)	Hatcol Corporation
HATCOL 2999	A polyol ester or pentaerythritol and short chain fatty acids (carrier oil)	Hatcol Corporation
HATCOL 3165	A polyol ester of dipentaerythritol and short chain fatty acids (carrier oil)	Hatcol Corporation
HATCOL 3371	A complexed polyol ester of trimethylol propane, adipic acid, caprylic acid, and capric acid (carrier oil)	Hatcol Corporation
HATCOL 5150	A polyol ester of dipentaerythritol and short chain fatty acids (carrier oil)	Hatcol Corporation
HELOXY 71	An aliphatic epoxy ester resin (carrier oil)	Hexion Specialty Chemicals, Inc., Houston,
HELOXY 505	An aliphatic epoxy ester resin (carrier oil)	Hexion Specialty Chemicals, Inc.

-continued

Glossary

Name	Description	Source
Lithium Stearate	A fatty acid salt (ionic dispersant)	Baerlocher USA, Cincinnati, OH
PEG Distearate	Poly(ethylene glycol) distearate having a number average molecular weight of about 930 (carrier oil/polymeric dispersant)	Aldrich Chemical Co., Milwaukee, WI
SOLPLUS 520	A polymeric dispersant	Noveon, Inc., a subsidiary of Lubrizol Corporation, Cleveland, OH
SOLSPERSE 24000	A polymeric dispersant	Noveon, Inc.
SOLSPERSE 39000	A polymeric dispersant	Noveon, Inc.
TONE 305	A polyol resulting from the addition reaction of caprolactone with trimethylol propane (carrier oil)	The Dow Chemical Company, Midland, MI
WA 30000	Aluminum oxide particles having a D_{50} of 0.25 μm	Fujimi Corporation
WA 4000	Aluminum oxide particles having a D_{50} of 3.0 μm	Fujimi Corporation
WA 500	Aluminum oxide particles having a D_{50} of 30 μm	Fujimi Corporation

[0045] "Sulfonated Bis(pentane dicaprolactone)", an ionic dispersant, was prepared as follows: To a reactor equipped with a mechanical stirrer, and vacuum was added 25 grams (0.476 equivalents) 1,5-pentane diol from Aldrich Chemical Co., Milwaukee, Wis., 54.3 grams (.476 equivalents) caprolactone from Aldrich Chemical Co., and 8.0 grams (0.054 equivalents) dimethyl-5-sodiosulfoisophthalate available from DuPont Chemicals, Wilmington, De.. The reactor contents were stirred and heated to 170° C. with a vacuum at 115 mm mercury. The reaction was complete after 4 hours and a sample was analyzed by infrared spectroscopy. The final product was a clear, low viscosity liquid with a theoretical sulfonate equivalent weight of 1342.

[0046] "iC8 Modified silica nanoparticles", a nonionic, inorganic dispersant, was prepared as follows: 61.42 grams BS1316 isooctyltrimethoxysilane (Wacker Silicones Corp., Adrian, Mich.), 1940 grams 1-methoxy-2-propanol and 1000 grams NALCO 2326 colloidal silica were combined in a 1 gallon glass jar. The mixture was shaken to ensure mixing and then placed in an oven at 80° C. overnight. The mixture was then dried in a flow through oven at 150° C. to produce a white particulate solid.

[0047] "HIMOD", a sulfonated polyol ionic dispersant, was prepared as follows: A reactor equipped with a mechanical stirrer, nitrogen purge, and distillation apparatus was charged with dimethyl-5-sodiosulfoisophthalate (42.6 grams, 0.144 moles, available from DuPont Chemicals, Wilmington, Del.), polyethylene glycol having a molecular weight of 400 (115.1 grams, 0.288 moles, available from Union Carbide Chemical and Plastics Co., Inc. (now The Dow Chemical Company, Midland, Mich.)), and polypropylene glycol having a molecular weight of 425 (122.3 grams, 0.288 moles, available from Aldrich Chemical Co., Milwaukee, Wis.), and xylene (75 grams). The reactor was slowly heated to 220° C. for about 1 hour to remove the xylene. Zinc acetate (0.2 gram) was then added to the reactor and the temperature was held at 220° C. for 4 hours with concomitant distillation of methanol from the reaction. The temperature was reduced to about 160° C. and 0.2 Torr (SI) vacuum was applied to the resulting mixture for 30 minutes. The contents were cooled to 120° C. under nitrogen to yield a clear, colorless polyol. The OH equivalent was determined to be 310 g/mole OH and the theoretical sulfonated equivalent weight was found to be 1882 grams polymer/mole sulfonated.

[0048] "TCPA HATCOL 3371", an ionic dispersant was prepared as follows: To a reactor equipped with a mechanical stirrer, and nitrogen purge was added 45 grams (0.0241 equivalents) HATCOL 3371 and 3.4 grams (0.0121 equivalents) tetrachlorophthalic anhydride. The reactor contents were stirred and heated to 150° C. with a constant nitrogen purge. The reaction was complete after 4 hours and a sample was analyzed by infrared spectroscopy. The final product was a brown, low viscosity liquid with a theoretical acid equivalent weight of 18,127.

[0049] "TONE 305 TCPA", an ionic dispersant, was prepared as follows: To a reactor equipped with a mechanical stirrer, and nitrogen purge was added 10 grams (0.1 equivalents) Tone 305 from Dow Chemical Company, and 1.0 grams (0.00355 equivalents) tetrachlorophthalic anhydride from Aldrich Chemical. The reactor contents were stirred and heated to 105° C. with a constant nitrogen purge. The reaction was complete after 4 hours and a sample was analyzed by infrared spectroscopy. The final product was a clear, low viscosity liquid with a theoretical acid equivalent weight of 3,100.

Sample Preparation

[0050] Except as noted in specific Examples, dispersant or mixture of dispersants was weighed into a watch glass. Any other surface active ingredients, if present, were also weighed onto the watch glass. Carrier oil(s), if present, was added to the dispersant(s) and the mixture was stirred with a metal spatula until the dispersant(s) was fully mixed into the carrier oil. Thermally conductive particles were added to the dispersant(s)/carrier oil mixture sequentially, starting with the smallest particle size distribution. Each of the thermally conductive particle distributions was dispersed into the dispersant(s)/carrier oil mixture with a metal spatula before adding the next distribution of thermally conductive particles. If necessary, the thermally conductive grease composition was heated in an oven (110° C.) to reduce the viscosity of the composition to facilitate mixing of the thermally conductive particles and/or subsequent additions of thermally conductive particles. The resultant thermally conductive greases were transferred into and stored in capped glass vials.

[0051] In cases where the thermally conductive particles was pre-dispersed, the amount of dispersant to be carried on the fine thermally conductive particle distribution was calculated. The amount of remaining dispersant necessary for the formulation was then determined and was weighed on to a watch glass. The remaining steps are identical to those described above.

EXAMPLES 1-64

[0052] The compositions of Examples 1-64 are shown in TABLE 1. TABLE 2 shows data resulting from the measurement of bulk conductivity and thermal impedance for selected Examples. TABLE 3 shows viscosity data for selected Examples.

TABLE 1

Example	Carrier Oil (g)	Dispersant (g)	Dispersant (g)	Particle (g) (D_{50}, μm)	Particle (g) (D_{50}, μm)	Particle (g) (D_{50}, μm)
Example 1	HATCOL 1106 (0.32); HATCOL 3371(0.32)	SOLSPERSE 39000 (0.36)	—	GC 20000 (2.12)	GC 4000 (2.97)	GC 400 (3.92)
Example 2	HATCOL 1106 (0.37); HATCOL 3371 (0.37)	SOLSPERSE 39000 (0.36)	—	GC 20000 (2.08)	GC 4000 (2.97)	GC 400 (3.88)
Example 3	HATCOL 1106 (0.42); HATCOL 3371 (0.42)	SOLSPERSE 39000 (0.35)	—	GC 20000 (2.07)	GC 4000 (2.91)	GC 400 (3.84)
Example 4	HATCOL 3371 (1.60)	SOLSPERSE 39000 (0.90)	—	GC 20000 (5.28)	GC 4000 (7.40)	GC 400 (9.81)
Example 5	HATCOL 3371 (0.74)	SOLSPERSE 39000 (0.36)	—	GC 20000 (2.08)	GC 4000 (2.93)	GC 400 (3.89)
Example 6	HATCOL 3371 (0.85)	SOLSPERSE 39000 (0.35)	—	GC 20000 (2.07)	GC 4000 (2.90)	GC 400 (3.82)
Example 7	—	SOLSPERSE 39000 (1.10)	—	GC 20000 (2.09)	GC 4000 (2.93)	GC 400 (3.90)
Example 8	HATCOL 1106 (0.37); HATCOL 3371 (0.37)	SOLSPERSE 39000 (0.27)	GAFAC RE 610 (0.09)	GC 20000 (2.10)	GC 4000 (2.93)	GC 400 (3.89)
Example 9	HATCOL 1106 (0.37); HATCOL 3371 (0.37)	SOLSPERSE 39000 (0.27)	HIMOD (0.09)	GC 20000 (2.09)	GC 4000 (2.94)	GC 400 (3.88)
Example 10	HATCOL 3371 (0.75)	SOLSPERSE 39000 (0.18)	GAFAC RE 610 (0.18)	GC 20000 (2.10)	GC 4000 (2.92)	GC 400 (3.87)
Example 11	HATCOL 3371 (0.74)	SOLSPERSE 39000 (0.27)	GAFAC RE 610 (0.09)	GC 20000 (2.09)	GC 4000 (2.92)	GC 400 (3.89)
Example 12	HATCOL 3371 (0.57)	SOLSPERSE 39000 (0.27)	TCPA HATCOL 3371 (0.27)	GC 20000 (2.09)	GC 4000 (2.94)	GC 400 (3.90)
Example 13	HATCOL 1106 (0.37); HATCOL 3371 (0.37)	SOLSPERSE 39000 (0.27)	Lithium Stearate (0.09)	GC 20000 (2.08)	GC 4000 (2.93)	GC 400 (3.89)
Example 14	HATCOL 3371 (0.15)	SOLSPERSE 39000 (0.08)	2,2' Bypyridylethylene bis-salicylimine (0.02)	GC 20000 (0.50)	GC 4000 (0.70)	GC 400 (0.93)
Example 15	HATCOL 3371 (0.15)	SOLSPERSE 39000 (0.08)	Ethylene bis-salycimine (0.02)	GC 20000 (0.49)	GC 4000 (0.69)	GC 400 (0.92)
Example 16	HATCOL 3371 (0.16)	SOLSPERSE 39000 (0.09)	BYK 361 (0.03)	GC 20000 (0.53)	GC 4000 (0.74)	GC 400 (0.98)
Example 17	HELOXY 71 (0.83)	SOLSPERSE 39000 (0.27)	—	GC 20000 (2.10)	GC 4000 (2.92)	GC 400 (3.87)
Example 18	HELOXY 71 (0.94)	SOLSPERSE 39000 (0.26)	—	WA 30000 (2.09)	WA 4000 (3.00)	WA 500 (3.83)

TABLE 1-continued

Example	Carrier Oil (g)	Dispersant (g)	Dispersant (g)	Particle (g) (D_{50}, μm)	Particle (g) (D_{50}, μm)	Particle (g) (D_{50}, μm)
Example 19	HATCOL 3371 (0.94)	SOLSPERSE 39000 (0.26)	—	WA 30000 (2.07)	WA 4000 (2.90)	WA 500 (3.83)
Example 20	TONE 305 (0.85)	SOLSPERSE 39000 (0.35)	—	GC 20000 (2.07)	GC 4000 (2.90)	GC 400 (3.83))
Example 21	TONE 305 (0.75)	SOLSPERSE 39000 (0.27)	Sulfonated Bis(pentane dicaprolactone) (0.09)	GC 20000 (2.09)	GC 4000 (2.94)	GC 400 (3.88)
Example 22	TONE 305 (0.85)	SOLSPERSE 39000 (0.26)	TCPA modified TONE 305 (0.09)	GC 20000 (2.07)	GC 4000 (2.90)	GC 400 (3.83)
Example 23	TONE 305 (0.85)	SOLSPERSE 39000 (0.26)	GAFAC RE 610 (0.09)	GC 20000 (2.07)	GC 4000 (2.91)	GC 400 (3.85)
Example 24	TONE 305 (0.75)	SOLSPERSE 39000 (0.36)	—	GC 20000 (2.08)	GC 4000 (2.93)	GC 400 (3.88)
Example 25	HATCOL 3371 (0.74)	SOLSPERSE 39000 (0.27)	GAFAC RE 610 (0.09)	GC 20000 (2.09)	GC 4000 (2.94)	GC 400 (3.90)
Example 26	HATCOL 3371 (0.74)	SOLSPERSE 39000 (0.27)	GAFAC RE 610 (0.09)	GC 20000 (2.09)	GC 4000 (2.92)	GC 400 (3.89)
Example 27	HATCOL 3371 (0.74)	SOLSPERSE 39000 (0.27)	GAFAC RE 610 (0.09)	GC 20000 (2.09)	GC 4000 (2.93)	GC 400 (3.88)
Example 28	HATCOL 3371 (0.74)	SOLSPERSE 39000 (0.27)	Sulfonated pentanediolcaprolactone (0.09)	GC 20000 (2.09)	GC 4000 (2.93)	GC 400 (3.89)
Example 29	HATCOL 3371 (0.74)	SOLSPERSE 39000 (0.36)	—	GC 20000 (2.09) (0.3)	GC 2000 (2.93) (9.0)	F180 SiC (3.88) (80)
Example 30	HATCOL 1106 (0.74)	SOLSPERSE 39000 (0.36)	—	GC 20000 (2.10)	GC 2000 (2.93)	F180 SiC (3.89)
Example 31	HATCOL 3371 (0.74)	SOLSPERSE 39000 (0.36)	—	GC 20000 (2.09)	GC 2000 (2.94)	F180 SiC (3.88)
Example 32	HATCOL 3371 (0.74)	SOLSPERSE 39000 (0.27)	GAFAC RE 610 (0.09)	GC 20000 (2.09)	GC 1200 (2.93)	F180 SiC (3.89)
Example 33	HATCOL 3371 (0.74)	SOLSPERSE 39000 (0.27)	PEG Distearate (0.09)	GC 20000 (2.10)	GC 2000 (2.93)	F180 SiC (3.88)
Example 34	HATCOL 3371 (0.74)	SOLSPERSE 39000 (0.36)	iC8 Modified silica nanoparticles (0.01)	GC 20000 (2.09)	GC 2000 (2.93)	F180 SiC (3.89)
Example 35	HATCOL 1106 (0.74)	SOLSPERSE 39000 (0.28)	GAFAC RE 610 (0.09)	GC 20000 (2.09)	GC 2000 (2.93)	F180 SiC 3.88)
Example 36	—	SOLSPERSE 39000 (0.80)	—	DP 1 (2.16) (0.25)	DP 2 (3.03) (3.0)	DP 2 (4.04) (30)
Example 37	HATCOL 2300 (0.25)	SOLSPERSE 39000 (0.55)	—	DP 1 (2.19) (0.25)	DP 2 (3.03) (3.0)	DP 2 (4.02) (30)
Example 38	HATCOL 2300 (0.52)	SOLSPERSE 39000 (0.28)	—	DP 1 (2.14) (0.25)	DP 2 (3.03) (3.0)	DP 2 (4.03) (30)
Example 39	HATCOL 2930 (0.52)	SOLSPERSE 39000 (0.28)	—	DP 1 (2.18) (0.25)	DP 2 (3.05) (3.0)	DP 2 (4.02) (30)
Example 40	HATCOL 3165 (0.52)	SOLSPERSE 39000 (0.28)	—	DP 1 (2.15) (0.25)	DP 2 (3.04) (3.0)	DP 2 (4.02) (30)
Example 41	HATCOL 3371 (0.52)	SOLSPERSE 39000 (0.28)	—	DP 1 (2.18) (0.25)	DP 2 (3.04) (3.0)	DP 2 (4.02) (30)

TABLE 1-continued

Example	Carrier Oil (g)	Dispersant (g)	Dispersant (g)	Particle (g) (D_{50}, µm)	Particle (g) (D_{50}, µm)	Particle (g) (D_{50}, µm)
Example 42	HATCOL 3371 (0.83)	SOLSPERSE 39000 (0.27)	—	GC 20000 (2.09)	GC 4000 (2.92)	GC 400 (3.89)
Example 43	HELOXY 71 (0.74)	SOLSPERSE 39000 (0.36)	—	DP 1 (2.10) (0.25)	DP 2 (2.93) (6.0)	DP 2 (3.89) (60)
Example 44 (1)	HELOXY 71 (0.52)	SOLSPERSE 39000 (0.28)	—	DP 2 (0.83) (0.1)	DP 2 (1.43) (1.0)	DP 2 (2.53) (9.0)
Example 45	HELOXY 71 (1.08)	SOLSPERSE 39000 (0.92)	—	DP 1 (5.40) (0.25)	DP 2 (7.58) (6.0)	DP 2 (10.0) (60)
Example 46	HATCOL 1106 (1.15)	SOLSPERSE 24000 (0.13)	—	DP 1 (3.55) (0.25)	DP 2 (6.50) (3.0)	DP 2 (11.0) (30)
Example 47 (2)	HATCOL 1106 (0.51)	SOLSPERSE 24000 (0.31)	—	DP 1 (2.54) (0.25)	DP 2 (4.66) (3.0)	DP 2 (7.94) (30)
Example 48 (2)	HATCOL 1106 (0.35)	SOLSPERSE 24000 (0.46)	—	DP 1 (2.53) (0.25)	DP 2 (4.67) (3.0)	DP 2 (7.96) (30)
Example 49	HATCOL 1106 (0.51)	SOLSPERSE 39000 (0.46)	—	DP 1 (2.39) (0.25)	DP 2 (4.69) (3.0)	DP 2 (7.94) (30)
Example 50 (2)	HATCOL 1106 (0.73)	SOLSPERSE 24000 (0.21)	—	DP 2 (2.14) (1.0)	DP 2 (2.99) (6.0)	DP 2 (3.97) (30)
Example 51 (2)	HELOXY 71 (0.74)	SOLSPERSE 24000 (0.21)	—	DP 2 (2.12) (1.0)	DP 2 (2.96) (6.0)	DP 2 (3.98) (30)
Example 52 (2)	HATCOL 1106 (0.74)	SOLSPERSE 24000 (0.25)	—	DP 1 (2.10) (0.5)	DP 2 (2.98) (6.0)	DP 2 (4.00) (45)
Example 53 (2)	HELOXY 71 (0.76)	SOLSPERSE 24000 (0.24)	—	DP 1 (2.10) (0.5)	DP 2 (2.97) (6.0)	DP 2 (3.98) (45)
Example 54 (2)	HELOXY 71 (0.63)	SOLSPERSE 24000 (0.04)	—	DP 1 (2.25) (0.25)	DP 2 (3.08) (3.0)	DP 2 (4.05) (30)
Example 55	HELOXY 71 (0.64)	SOLSPERSE 39000 (0.16)	—	DP 1 (2.19) (0.25)	DP 2 (3.06) (3.0)	DP 2 (4.05) (30)
Example 56	HELOXY 71 (0.45)	SOLSPERSE 39000 (0.15)	—	DP 1 (1.78) (0.25)	DP 2 (3.04) (3.0)	DP 2 (4.63) (30)
Example 57	HELOXY 71 (0.55)	SOLSPERSE 39000 (0.15)	—	DP 1 (1.90) (0.25)	DP 2 (3.02) (3.0)	DP 2 (4.28) (30)
Example 58	HATCOL 2949 (0.64)	SOLSPERSE 39000 (0.17)	—	DP 1 (2.17) (0.25)	DP 2 (3.02) (3.0)	DP 2 (4.03) (30)
Example 59	HATCOL 2300 (0.64)	SOLSPERSE 39000 (0.17)	—	DP 1 (2.19) (0.25)	DP 2 (3.02) (3.0)	DP 2 (4.02) (30)
Example 60	HATCOL 2999 (0.64)	SOLSPERSE 39000 (0.17)	—	DP 1 (2.16) (0.25)	DP 2 (3.04) (3.0)	DP 2 (4.01) (30)
Example 61	HATCOL 5150 (0.64)	SOLSPERSE 39000 (0.17)	—	DP 1 (2.19) (0.25)	DP 2 (3.03) (3.0)	DP 2 (4.03) (30)
Example 62	HELOXY 505 (0.63)	SOLSPERSE 39000 (0.17)	—	DP 1 (2.14) (0.25)	DP 2 (3.03) (3.0)	DP 2 (4.04) (30)
Example 63	HELOXY 71 (0.78)	SOLSPERSE 39000 (0.17)	—	GC 8000 (2.12)	GC 2000 (2.98)	F180 SiC (3.96)

TABLE 1-continued

Example	Carrier Oil (g)	Dispersant (g)	Dispersant (g)	Particle (g) (D_{50}, μm)	Particle (g) (D_{50}, μm)	Particle (g) (D_{50}, μm)
Example 64	HELOXY 71 (0.70)	SOLSPERSE 39000 (0.20)	—	DP 1 (1.91) (0.25)	GC 4000 (2.67)	GC 700 (3.54)

(1) Example 44 contained a 4th thermally conductive particle: DP 2, (4.41 grams), (60 μm).
(2) Examples 46–48 and 50–54 used 0.25, 0.50, or 1.0 μm pre-dispersed diamond particles prepared according to the Milling Procedure and Sample Preparation described above.

[0053]

TABLE 2

Example	Bulk Conductivity (W/m-K)	Thermal Impedance at 100 μm meter bar gap (° C.-cm^2/W)
1	3.71	0.497
2	3.50	0.542
3	2.86	0.555
4	4.18	0.518
5	3.53	0.476
6	3.21	0.602
7	4.19	0.355
8	3.74	0.520
9	3.42	0.548
10	3.84	0.431
11	4.24	0.444
12	3.52	0.425
13	3.71	0.528
14	3.78	0.464
15	3.77	0.532
16	3.58	0.555
17	4.24	0.644
18	3.86	0.547
19	3.15	0.482
20	3.54	0.616
21	3.62	0.622
22	4.10	0.608
23	3.71	0.638
24	3.91	0.580
25	3.95	0.545
26	3.93	0.63
27	3.44	0.605
28	3.44	0.604
29	4.45	0.652
30	3.49	0.628
31	3.84	0.625
32	3.65	0.582
33	3.28	0.507
34	3.01	0.569
35	3.63	0.595
36	5.01	0.409
37	4.92	0.389
38	4.58	0.451
39	3.71	0.464
40	4.47	0.514
41	4.23	0.451
42	2.73	0.412
43	3.52	0.662
44	5.88	0.491
45	5.62	0.519
46	4.35	0.473
47	6.31	0.421
48	6.80	0.388
49	6.12	0.395
50	3.18	0.821
51	3.33	0.728
52	2.78	0.871
53	2.96	0.839
54	4.11	0.535
55	4.00	0.403

TABLE 2-continued

Example	Bulk Conductivity (W/m-K)	Thermal Impedance at 100 μm meter bar gap (° C.-cm^2/W)
56	5.22	0.351
57	4.92	0.372
58	2.44	0.398
59	3.35	0.514
60	3.62	0.562
61	3.56	0.596
62	4.18	0.501
63	4.24	0.644
64	2.73	0.412
CE 1	2.49	0.766
CE 2	2.54	0.665

CE 1 = ShinEtsu G751, Sample 1
CE 2 = ShinEtsu G751, Sample 2

[0054]

TABLE 3

Example	0.5 mm Gap η (mPa · s) @ 25° C. & 1.25/sec Shear Rate	0.25 & 0.5 mm Gap Ave. η (mPa · s) @ 125° C. & 1.25/sec Shear Rate	0.25 mm Gap η (mPa · s) @ 125° C. & 1.25/sec Shear Rate
26	—	4.4E+04	5.8E+04
28	—	1.1E+06	1.0E+06
30	2.7E+06	—	1.3E+04
31	—	9.2E+04	7.9E+04
32	—	2.5E+04	3.8E+04
35	—	—	1.7E+04
43	—	4.2E+04	2.9E+04
44	—	—	2.4E+05
45	4.4E+06	—	—
CE	1.2E+06	4.3E+05	3.1E+05

CE = ShinEtsu G751

[0055] Foreseeable modifications and alterations of this invention will be apparent to those skilled in the art without departing from the scope and spirit of this invention. This invention should not be restricted to the embodiments that are set forth in this application for illustrative purposes.

What is claimed is:

1. A thermally conductive grease comprising:

0 to about 49.5 weight percent of carrier oil;

about 0.5 to about 25 weight percent of at least one dispersant; and

at least about 49.5 weight percent of thermally conductive particles, wherein the thermally conductive particles comprise a mixture of at least three distributions of thermally conductive particles, each of the at least three distributions of thermally conductive particles having an average (D_{50}) particle size which differs from the other distributions by at least a factor of 5.

2. The thermally conductive grease of claim 1 wherein each of the at least three distributions of thermally conductive particles having an average (D_{50}) particle size which differs from the others by at least a factor of 7.5.

3. The thermally conductive grease of claim 1 wherein each of the at least three distributions of thermally conductive particles having an average (D_{50}) particle size which differs from the others by at least a factor of 10.

4. The thermally conductive grease of claim 1 wherein the thermally conductive particles comprise materials selected from the group consisting of diamond, silicon carbide, alumina, boron nitride (hexagonal or cubic), boron carbide, silica, graphite, amorphous carbon, polycrystalline diamond, aluminum nitride, aluminum, silver, and combinations thereof.

5. The thermally conductive grease of claim 1 wherein the dispersant comprises a dispersant selected from the group consisting of nonionic, dispersants, polymeric dispersants, ionic dispersants, inorganic dispersants, and combinations thereof.

6. The thermally conductive grease of claim 1 wherein carrier oil is present in an amount of from about 0.5 to about 20 weight percent.

7. The thermally conductive grease of claim 1 wherein one of the at least three distributions of thermally conductive particles has an average particle size that ranges from about 0.02 to about 5 micrometers.

8. The thermally conductive grease of claim 1 wherein one of the at least three distributions of thermally conductive particles has an average particle size that ranges from about 0.10 to about 50.0 micrometers.

9. The thermally conductive grease of claim 1 wherein one of the at least three distributions of thermally conductive particles has an average particle size that ranges from about 0.50 to about 500 micrometers.

10. The thermally conductive grease of claim 1 wherein the at least one dispersant comprises an ionic dispersant and a polymeric dispersant.

11. The thermally conductive grease of claim 1 further comprising a fourth distribution of thermally conductive particles.

12. The thermally conductive grease of claim 1 wherein the thermally conductive particles comprise a mixture of diamond and silicon carbide particles.

13. The thermally conductive grease of claim 1 wherein the at least three distributions of thermally conductive particles are essentially non-overlapping.

14. The thermally conductive grease of claim 1 wherein the at least three distributions of thermally conductive particles have a minimal overlap.

15. A method of making a thermally conductive grease comprising the steps of:

providing carrier oil, dispersant, and thermally conductive particles of claim 1;

mixing the carrier oil and dispersant together; and

mixing the thermally conductive particles sequentially, finest to largest average particle size into the carrier oil and dispersant mixture.

16. The method of claim 15 wherein the thermally conductive particles are pretreated with dispersant prior to mixing the thermally conductive particles into the carrier oil and dispersant mixture.

17. A method of making a thermally conductive grease comprising the steps of:

providing carrier oil, dispersant, and thermally conductive particles of claim 1;

mixing the thermally conductive particles together;

mixing the carrier oil and dispersant together; and

mixing the mixed thermally conductive particles with the carrier oil and dispersant mixture.

18. The method of claim 17 wherein the thermally conductive particles are pretreated with dispersant prior to mixing the thermally conductive particles into the carrier oil and dispersant mixture.

19. A microelectronic package comprising:

a substrate;

at least one microelectronic heat source attached to the substrate; and

the thermally conductive grease of claim 1 on the at least one microelectronic heat source.

20. The microelectronic package of claim 18 further comprising a heat spreader and the thermally conductive grease is between the microelectronic heat source and the heat spreader.

21. The microelectronic package of claim 19 further comprising a heat dissipation device wherein thermally conductive grease is between the heat spreader and the heat dissipation device.

22. The thermally conductive grease of claim 11 wherein the thermally conductive particles comprise materials selected from the group consisting of diamond, silicon carbide, alumina, boron nitride (hexagonal or cubic), boron carbide, silica, graphite, amorphous carbon, polycrystalline diamond, aluminum nitride, aluminum, silver, and combinations thereof.

23. The thermally conductive grease of claim 1 wherein the thermally conductive particles in at least one of the at least three distributions of thermally conductive particles comprises a mixture of at least two different types of thermally conductive particles.

24. The thermally conductive grease of claim 1 wherein the thermally conductive particles in at least one of the at least three distributions of thermally conductive particles contain thermally conductive particles of a type different from the thermally conductive particles in the other particle distributions.

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