A silicone adhesive composition is provided having a latent crosslinker that provides increased tack and peel performance.
HIGH PERFORMANCE ORGANOSILICON ADHESIVE
WITH LATENT CROSSLINKER

CROSS REFERENCE TO RELATED APPLICATIONS
[001] This application claims the priority date of PRC (China) Patent Application No. 201410855687.7, filed December 31, 2014, and claims priority to U.S. Provisional Patent Application No. 62/103,290, filed January 14, 2015, the disclosures of which are incorporated by reference herein in their entirety.

FIELD OF THE INVENTION
[002] The invention relates generally to a high performance organosilicon pressure sensitive adhesive tape with high initial tack and peel strength.

BACKGROUND OF THE INVENTION
[003] Commercially available organosilicon-based pressure sensitive adhesives ("PSAs") are known for their superior stability characteristics compared to organic polymer-based pressure sensitive adhesives and hence, are commonly employed for high temperature applications up to 260 °C. To maintain good adhesion and peel strength at high temperature, the adhesive film requires increased degree of crosslinking. High degree of crosslinking often results in increased film modulus and reduced initial tack performance at room temperature. It can be challenging in the field to synergistically achieve both high initial tack performance and good peel strength over a range of temperatures.
[004] Molecular design techniques are known to increase high temperature adhesion. Substitution of methyl groups for phenyl groups in the polydimethylsiloxane backbone of a silicone based pressure sensitive adhesive resin may provide some improvement but at a significant cost increase of the material for manufacture.
[005] Typical organosilicon-based pressure sensitive adhesives require a crosslinking agent that initiates crosslinking during drying of the pressure sensitive adhesive. Decreasing the amount of crosslinking agent decreases the degree of crosslinking, hence the good initial tack at room temperature. Increasing the amount of crosslinking agent increases peel strength but results in a loss of tack at room temperature. There is a need in the art to improve lower temperature initial tack while maintaining high temperature peel strength.

BRIEF SUMMARY OF THE INVENTION

[006] The present invention provides a high performance organosilicon adhesive composition containing a first crosslinker and a second latent crosslinker. A two-step crosslinking method is disclosed that provides an organosilicon pressure sensitive adhesive composition with good low temperature initial tack and increased peel strength at high temperature. Addition of a second latent crosslinker to the organosilicon pressure sensitive adhesive composition can be activated during post curing, for example, during tape drying, wherein the second latent crosslinker reacts at a higher temperature than the first crosslinker. The two-step crosslinking method provides an adhesive bond with good initial tack and an increased degree of crosslinking at high temperature resulting in increased peel strength and overall adhesion.

[007] In one embodiment an adhesive composition comprises an organosilicon-based pressure sensitive adhesive, a first crosslinker, and a second latent crosslinker, wherein the second latent crosslinker has a higher initiation temperature than the first crosslinker.

[008] In another embodiment the adhesive composition comprises polydialkylsiloxane, polyalkylarylsiloxane, polydiarylsiloxane, polyalkylalkenylsiloxane, or combinations thereof, or copolymers thereof.

[009] In another embodiment the adhesive composition comprises a DT or MQ resin.
[010] In another embodiment the adhesive composition comprises a solvent, wherein the solvent is chloroform, methylene chloride, 1,2-dichloroethane, benzene, toluene, xylene, heptane, hexane, isoparaffins, petroleum ether, 2,6-dichlorotoluene, or combinations thereof.

[011] In another embodiment the first crosslinker of the adhesive composition is benzoyl peroxide, 2,4-dichlorobenzoyl peroxide, or combinations thereof.

[012] In another embodiment the second latent crosslinker of the adhesive composition is cumyl hydrogen peroxide (CHP), dicumyl peroxide (DCP), tert-butyl hydrogen peroxide (TBHP), ditert-butyl peroxide (DTBP), 2,5-dimethyl-2,5-di(t-butylperoxy)hexane (DPBH), 2,5-di(t-butylperoxy)-2,5-di(tert-butylperoxy)-2,5-dimethyl-3-hexyne (LQ-DYBP), tert-butyl peroxybenzoate (TBPB), or combinations thereof.

[013] In another embodiment the $\Delta T$ between the first crosslinker and the second latent crosslinker of the adhesive composition is greater than 60 °C.

[014] In another embodiment the $\Delta T$ between the first crosslinker and the second latent crosslinker of the adhesive composition is greater than 80 °C.

[015] In another embodiment the $\Delta T$ between the first crosslinker and the second latent crosslinker of the adhesive composition is greater than 100 °C.

[016] In another embodiment an adhesive construct comprises an organosilicon-based pressure sensitive adhesive, a first crosslinker, a second latent crosslinker; and a backing, wherein the second latent crosslinker has a higher initiated temperature than the first crosslinker.

[017] In another embodiment the adhesive construct comprises polydialkylsiloxane, polyalkylarylsiloxane, polydiarylsiloxane, polyalkylalkenylsiloxane, or combinations thereof, or copolymers thereof.

[018] In another embodiment the adhesive construct comprises a DT or MQ resin.

[019] In another embodiment the adhesive construct comprises a solvent, wherein the solvent is chloroform, methylene chloride, 1,2-dichloroethane,
benzene, toluene, xylene, heptane, hexane, isoparaffins, petroleum ether, 2,6-
dichlorotoluene, or combinations thereof.

[020] In another embodiment the first crosslinker of the adhesive construct is benzoyl peroxide, 2,4-dichlorobenzoyl peroxide, or combinations thereof.

[021] In another embodiment the second latent crosslinker of the adhesive construct is cumyl hydrogen peroxide (CHP), dicumyl peroxide (DCP), tert-butyl hydrogen peroxide (TBHP), ditert-butyl peroxide (DTBP), 2,5-dimethyl-2,5-di(t-butylperoxy)hexane (DPBH), 2,5-Di(tert-butylperoxy)-2,5-dimethyl-3-hexyne (LQ-DYBP), tert-butyl peroxycaprylate (TBPB), or combinations thereof.

[022] In another embodiment the ΔT between the first crosslinker and the second latent crosslinker of the adhesive construct is greater than 60 °C.

[023] In another embodiment the ΔT between the first crosslinker and the second latent crosslinker of the adhesive construct is greater than 80 °C.

[024] In another embodiment the ΔT between the first crosslinker and the second latent crosslinker of the adhesive construct is greater than 100 °C.

[025] In another embodiment the backing of the adhesive construct is paper, cloth, glass cloth, fiber glass, aluminum, silicone rubber, silicone resin-treated glass cloth, polyethylene terephthalate (PET), polytetrafluoroethylene (PTFE), polychlorotrifluoroethylene (PCTFE), polyvinyl chloride (PVC), polypropylene (PP), polyethylene (PE), polyimide film, aromatic polyamide paper, or combinations thereof.

[026] In another embodiment a method to prepare a bonded product from an adhesive construct comprises an organosilicon-based pressure sensitive adhesive, a first crosslinker, a second latent crosslinker, and a backing, wherein the second latent crosslinker has a higher initiated temperature than the first crosslinker comprising the steps of:

a) diluting the organosilicon-based pressure sensitive adhesive in toluene to a solid content of 30-60% and mixing until homogenous;
b) adding 0.5-5% of a first crosslinker and adding 0.5-5% of a second latent crosslinker directly or dissolved in toluene to the diluted organosilicon-based pressure sensitive adhesive and continue mixing to provide an adhesive composition;
c) placing a coating table in a hood and placing a primed backing substrate on top of the coating table with primed side facing up;
d) measuring appropriate amount of the adhesive composition from step b) and preparing a four-sided coater;
e) coating the primed side of the backing substrate with the adhesive composition to provide the adhesive construct;
f) place the adhered construct in a first oven which has been heated to 90 °C for 2 minutes and then transfer the adhered construct to a second oven which has been heated to 180 °C for 2 minutes and then remove and allow the adhered construct to cool to provide the bonded product.

[027] While multiple embodiments are disclosed, still other embodiments of the present invention will become apparent to those skilled in the art from the following detailed description. As will be apparent, the invention is capable of modifications in various obvious aspects, all without departing from the spirit and scope of the present invention. Accordingly, the detailed descriptions are to be regarded as illustrative in nature and not restrictive.

BRIEF DESCRIPTION OF THE DRAWINGS

[028] FIG. 1 shows the peeling strength of commercial PSA.
[029] FIG. 2 shows the peeling strength of compositions described in the current embodiments.
[030] FIG. 3 shows the rolling ball initial tack test results of compositions described in the current embodiments.
DETAILED DESCRIPTION

[031] In the specification and in the claims, the terms "including" and "comprising" are open-ended terms and should be interpreted to mean "including, but not limited to. . ." These terms encompass the more restrictive terms "consisting essentially of" and "consisting of."

[032] It must be noted that as used herein and in the appended claims, the singular forms "a", "an", and "the" include plural reference unless the context clearly dictates otherwise. As well, the terms "a" (or "an"), "one or more" and "at least one" can be used interchangeably herein. It is also to be noted that the terms "comprising", "including", "characterized by" and "having" can be used interchangeably.

[033] Unless defined otherwise, all technical and scientific terms used herein have the same meanings as commonly understood by one of ordinary skill in the art to which this invention belongs. All publications and patents specifically mentioned herein are incorporated by reference in their entirety for all purposes including describing and disclosing the chemicals, instruments, statistical analyses and methodologies which are reported in the publications which might be used in connection with the invention. All references cited in this specification are to be taken as indicative of the level of skill in the art. Nothing herein is to be construed as an admission that the invention is not entitled to antedate such disclosure by virtue of prior invention.

[034] The present invention relates to organosilicon pressure sensitive adhesive compositions. More particularly the present invention relates to pressure sensitive compositions which are prepared by heating a mixture of a silicon-based pressure sensitive adhesive, a solvent, a first crosslinker, and a second latent crosslinker, wherein the second latent crosslinker has a higher initiation temperature than the first crosslinker.

[035] Conventional pressure sensitive adhesives comprise as main ingredients, natural rubber, a synthetic rubber, a polyvinyl alkyl ether, a polyacrylate or the
like, and as subsidiary ingredients, additives such as a tackifier and plasticizer. They are predominantly so-called solution-type pressure-sensitive adhesives where an organic solvent is selected from aromatic solvents, esters, ketones and the like, and are excellent in adhesion, applicability, drying property (curability), water resistance and the like.

[036] The organosilicon-based pressure sensitive adhesives of the current embodiments are either typically produced by either blending or condensing together an organosilicon resin and an organosilicon polymer. Typical diorganosiloxane compositions comprise polydimethylsiloxane or polymethylphenylsiloxane; copolymers of dimethylsiloxane and diphenylsiloxane units; copolymers of dimethylsiloxane and methylphenylsiloxane units; terpolymers of dimethylsiloxane, methylvinylsiloxane units and diphenylsiloxane units, and the like. Other polymers may include polyethylmethylsiloxane, polydiethylsiloxane, polyethylphenylsiloxane, polymethylvinylsiloxane, polydiphenylsiloxane, or copolymers thereof.

[037] The organosilicon-based pressure sensitive adhesives of the current embodiments may additionally comprise a DT resin or a MQ resin that may be included during condensation to provide highly crosslinked polysiloxane networks. DT resins are formed from D (Me₂SiO₂) and T (Me₃SiO₃) units and MQ resins are formed from M (Me₃SiO) and Q (SiO₄) units. Other combinations (MDT, MTQ, and QDT) may also be included in the current embodiments.

[038] The homo and copolymers can be blocked at the ends, e.g., with triorganosiloxy units, such as trimethylsiloxane, dimethylvinylsiloxane, dimethylphenylsiloxane units, and the like. End groups may also contain hydroxyl-terminated polydiorganosiloxane. Alternative organo substituents may be independently selected from a monovalent hydrocarbon or halohydrocarbon radical having from 1 to 20 carbon atoms or an alkenyl radical. The monovalent hydrocarbon radicals free of aliphatic unsaturation include alkyl radicals such as methyl, ethyl, propyl, pentyl, hexyl, octyl, undecyl or octadecyl, cycloaliphatic...
radicals such as cyclopentyl and cyclohexyl, aryl radicals such as phenyl, tolyl, xylyl, benzyl, naphthyl, or 2-phenylethyl, and halogenated hydrocarbon radicals such as 3-chloropropyl, dichlorophenyl, and trifluoroalkyl derivatives. The alkenyl radicals include vinyl, allyl, butenyl, hexenyl, cyclohexenyl and beta-cyclohexenylethyl.

[039] Preferably the organosilicon-based pressure sensitive adhesives are those acquired by commercial suppliers. The organosilicon-based pressure sensitive adhesive materials may be supplied as premixed solutions or as weighed solids that can be mixed thereafter. Commercial organosilicon-based pressure sensitive adhesives include Dow Corning® Q2-7735, Q2-7406, Q2-7566, 7268, 7355, 7356, 7358, 7388, 280A Adhesives; Shin-Etsu KRT-001, KRT-002, KRT-003, KRT-006, KRT-025, KRT-026, TSV-100, TSV-250; Momentive PSA518, SCA1000; Momentive SilGrip® PSA345SM, PSA529, PSA510, PSA590, PSA593, PSA610, PSA6573A, PSA6574, PSA6754A, PSA915; and Bluestar Silicones Silolease® PSA 400, PSA 401, PSA 408, and PSA 418; and Wacker SILRES® 64558 VP. Generally the ratios of the two or more organosilicon-based pressure sensitive materials (on a dry weight basis) ranges can be from 1:100 to 1:1 on a weight percent basis and all ranges there between, such as for example, 1:5, 1:10, 1:20, 1:30, 1:40; 1:50, 1:60, 1:70, 1:80, 1:90 and all values there between.

[040] The compositions of the current embodiments include at least two or more curing agents or crosslinkers. The curing of an organosilicon-based pressure sensitive adhesive refers to the toughening or hardening of the material by crosslinking of the polymer chains. Crosslinking may be achieved through the use of a radical initiator to induce polymerization. The radical initiators or crosslinkers of the current embodiments are used each in a range of 0.5% to 5% weight percentage of the total weight of the adhesive composition. Ratios of the crosslinkers can be from 1:100 to 1:1 on a weight percent basis and all ranges there between, such as for example, 1:5, 1:10, 1:20, 1:30, 1:40; 1:50, 1:60, 1:70,
1:80, 1:90 and all values there between. Preferably the first crosslinkers are benzoyl peroxide (BPO) and 2,4-dichlorobenzyol peroxide. 2,4-dichlorobenzyol peroxide is available as Cadox® TS 50 or Cadox® TDP from Akzo Chemie of America, Noury Chemical Division. Benzoyl peroxide is available as Lucidol®98 from Elfatochem North America, Cadox® BFF 50 powder or BP 55 paste from Akzo Chemie of America, Noury Chemical Division.

[041] In certain embodiments, the presence of an expensive platinum catalyst is excluded from the first crosslinker, the second crosslinker, or both.

[042] The second crosslinker may be referred to as a latent crosslinker. Latency of the second crosslinker in the current embodiments may refer to a chemical property of the second crosslinker in comparison to the first crosslinker. The second latent crosslinker may be chemically more stable than first traditional crosslinker. An increase in chemical stability of the second latent crosslinker may result in a higher decomposition temperature or initiating temperature. The higher initiating latent crosslinker used in the current embodiments include but are not limited to, cumyl hydrogen peroxide (CHP), dicumyl peroxide (DCP), tert-butyl hydrogen peroxide (TBHP), ditert-butyl peroxide (DTBP) or combinations thereof. The half-life of the second crosslinker may be greater than the half-life of the first crosslinker. The half-life of CHP is 260 °C for 1 min; DCP is 170 °C for 1 min; TBHP is 264 °C for 6 min; DTBP is 193 °C for 1 min; 2,5-dimethyl-2,5-di(t-butylperoxy)hexane (DPBH) is 196 °C for lmin; 2,5-di(t-tert-butylperoxy)-2,5-dimethyl-3-hexyne (LQ-DYBP) is 191 °C at lmin; and tert-butyl peroxybenzozate (TBPB) is 160 °C for lmin. The ΔT between the first and second crosslinkers is preferably greater than 60 °C, preferably greater than 80 °C, and most preferably greater than 100 °C. ΔT is intended to refer to the difference or range between the activation temperature between the first crosslinker and the second crosslinker. Typically, the first crosslinker is activated from about 90 °C to about 130 °C to effect crosslinking. The second crosslinker is then activated from 130 °C to about 260 °C to effect further crosslinking.
In preferred embodiments, the commercially obtained organosilicon-based pressure sensitive adhesive is mixed together with the first crosslinker and the second latent crosslinker. A conventional solvent may be used to dilute or further dilute the pressure sensitive adhesive and/or the first crosslinker and/or the second latent crosslinker prior to mixing and/or after mixing. Dilutions may be performed using a conventional solvent. Preferably the conventional solvent is an aromatic hydrocarbon like those used with organosilicon-based pressure sensitive adhesives acquired from commercial suppliers. Typically, the conventional solvents from commercial suppliers may be toluene or xylenes. The choice of dilution solvent may depend on boiling point, hydrophobicity, and/or polarity.

Generally the solids content of the adhesive mixture of adhesive and crosslinkers can be any value. In particular, the solids content can be adjusted from about 30 weight percent to about 60 weight percent and can be directly applied to a surface without further treatment. Optionally, the composition can be gently heated to help effect dissolution of one or more of the components. The gentle heating would be conducted at a temperature below that which would initiate the decomposition of the initiator(s). This is just one embodiment to prepare a suitable tape.

The adhesive mixture of adhesive and crosslinkers may be heated to effect interaction between the ingredients to permit homogeneity of the solution and to aid in the removal of the conventional solvent. Heating for one-half to six hours is generally sufficient to produce optimum tack and cohesive strength in the final adhesive product. The tack and cohesive strength may be readily determined by removing samples periodically from the mixture to establish whether a suitable level of initial tackiness and cohesive strength has been attained. The adhesive mixture may be then diluted again to provide desired consistency for various applications.

After a material of suitable initial tackiness and cohesive strength has been obtained, for example in the preparation of an adhesive tape, the
organosilicon-based pressure sensitive adhesive of the current embodiments may be dissolved in a solvent such as an aromatic hydrocarbon, or a halogenated hydrocarbon, such as chloroform, methylene chloride, 1,2-dichloroethane, or the like, preferably an aromatic or alkane hydrocarbon, such as benzene, toluene, xylene, heptane, hexane, or the like. The organosilicon-based pressure sensitive adhesive of the current embodiments may have a convenient solids content, for example, from about 20% to about 70%, and preferably from about 30% to about 60%.

[047] The organosilicon-based pressure sensitive adhesive solution may then advantageously be applied to tapes, sheets, or cloths and the like, by various means, such as brushing, spraying, knife coating, etc.

[048] Another important aspect of the current embodiments is to provide an organosilicon-based pressure sensitive adhesive applied to a surface of a backing substrate. The organosilicon-based pressure sensitive adhesive tapes, as well as sheets, etc., according to the present embodiments, may be obtained by applying the compositions, especially preferably as solvent solutions of the compositions, to various coherent unitary backings, such as paper, cloth, glass cloth, aluminum, fiber glass, silicone rubber, silicone resin-treated glass cloth, polyethylene terephthalate (PET), polytetrafluoroethylene (PTFE), polychlorotrifluoroethylene (PCTFE), polyimide film, aromatic polyamide paper, polyvinyl chloride (PVC), polypropylene (PP), polyethylene (PE) etc., or combinations thereof.

[049] Generally, the applied wet coatings are about 50 to 350 μη thick and after solvent removal the dried coatings are about 20 to 100 μη. In order to prevent adhesion of the pressure-sensitive adhesive to its own backing if it should be rolled up into cylinders or tapes, sheets, etc., a suitable anti-blocking or release coating or sheet may be interspersed between the surface of the pressure-sensitive adhesive and the adjacent uncoated surface of the backing for the pressure-sensitive adhesive. Those skilled in the art will be able to select appropriate anti-
blocking materials, and will be well aware of the conventional details of tape manufacture.

[050] The invention will be further described with reference to the following non-limiting Examples. It will be apparent to those skilled in the art that many changes can be made in the embodiments described without departing from the scope of the present invention. Thus the scope of the present invention should not be limited to the embodiments described in this application, but only by embodiments described by the language of the claims and the equivalents of those embodiments.

[051] Commercial PSAs used in the current embodiments were used as received from suppliers. Table 1 discloses the commercial PSAs utilized and their respective solid content as received.

[052] Table 1. Commercial organosilicon PSA.

<table>
<thead>
<tr>
<th>Suppliers</th>
<th>Grade</th>
<th>Solid content (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dow Corning</td>
<td>7358</td>
<td>55-58</td>
</tr>
<tr>
<td></td>
<td>7268</td>
<td>58-62</td>
</tr>
<tr>
<td></td>
<td>7388</td>
<td>55-58</td>
</tr>
<tr>
<td>Shin-Etsu</td>
<td>KRT-001</td>
<td>60</td>
</tr>
<tr>
<td></td>
<td>KRT-003</td>
<td>60</td>
</tr>
<tr>
<td></td>
<td>KRT-026</td>
<td>60</td>
</tr>
<tr>
<td>Momentive</td>
<td>PSA 529</td>
<td>55</td>
</tr>
<tr>
<td></td>
<td>PSA 510</td>
<td>55</td>
</tr>
<tr>
<td></td>
<td>PSA 6754A</td>
<td>-</td>
</tr>
<tr>
<td>Bluestar</td>
<td>PSA 400</td>
<td>55</td>
</tr>
<tr>
<td></td>
<td>PSA 408</td>
<td>60</td>
</tr>
</tbody>
</table>

[053] PTFE coated fiber glass was used as the backing material during experimentation. Commercial backing material SG25-03 and SG35-05 were used as received from Saint-Gobain having a thickness of 3 mil and 5 mil respectively. SG25-03 was used in the following examples. One "mil" is equal to one thousandths of an inch.

[054] Table 2. lists the PSA formulations prepared for the following examples.

[055] Table 2. Formulation.
Formulation | Si-PSA (wt%) | Crosslinker | Feed ratio (wt%)  
--- | --- | --- | ---  
0 | 1:1 PSA 400/408 | BPO | 2  
1 | 1:1 PSA 400/408 | BPO/CHP | 1:1  
2 | 1:1 PSA 400/408 | BPO/DCP | 1:1  
3 | 1:1 PSA 400/408 | BPO/TBHP | 1:1  
4 | 1:1 PSA 400/408 | BPO/DTBP | 1:1

[056] Example 1.

[057] Peel strength at 260 °C of commercial samples.

[058] Peeling strength at 260 °C has been validated as one of the critical technical qualities to evaluate high temperature performance. In addition to the adhesive tape made from formulation 0, additional commercial organosilicon PSA tapes (SG25-03US, SG25-03India, Nitto 973UL, Chukoh, and Changfeng) were also screened to establish performance benchmarks. High temperature peel strength results noted in FIG. 1 show that the performance of Saint-Gobain's products are comparable to that of Chukoh, but both are inferior to that of Nitto 973UL. Nitto 973UL was selected as the benchmark sample.

[059] Example 2.

[060] Peel strength at 260 °C of benchmark versus formulated products.

[061] Peeling strength is an important contributing factor to overall tape adhesion. The higher the peeling strength of the adhesive tape at high temperature, the longer the lifetime of the adhesive bond. FIG. 2 results of peeling strength at 260 °C show that second latent crosslinkers DCP, TBHP, and DTBP of formulations 2-4 provided improved peel strength over formulation 0 and benchmark Nitto 973UL. Surprisingly, formulation 2 provides twice the peeling performance compared to Nitto 973UL.

[062] Example 3.

[063] Rolling ball initial tack test.

[064] The method of oblique plane by rolling ball test at 30° is used to measure the initial tack of an adhesive and its ability to quickly adhere to a substrate. It is normally consistent with the perception of stickiness by touch. Here it is used to
test if the initial tack remains good where a latent crosslinker was used to enhance the peeling performance. FIG. 3 demonstrates that formulations 1 through 4 have good initial tack in comparison to Nitto 973UL and SG25-03US.


[066] Holding power test.

A holding power test was conducted at 260 °C to evaluate the cohesion strength of the adhesive film. The PSA tapes are adhered to a stainless steel panel by an area of 25.4 mm x 25.4 mm and placed in a 260 °C oven. A 1 kg weight is hung from the tape for 24 h and examined for tape displacement. A displacement of less than 1 mm is recorded as a pass.

[067] Table 3. Holding power test results.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Results</th>
<th>Displacement (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitto 973UL</td>
<td>Pass</td>
<td>0</td>
</tr>
<tr>
<td>SG25-03US</td>
<td>Pass</td>
<td>0</td>
</tr>
<tr>
<td>0</td>
<td>Pass</td>
<td>0</td>
</tr>
<tr>
<td>1</td>
<td>Fail</td>
<td>25+</td>
</tr>
<tr>
<td>2</td>
<td>Pass</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>Fail</td>
<td>2</td>
</tr>
<tr>
<td>4</td>
<td>Fail</td>
<td>3</td>
</tr>
</tbody>
</table>

[068] The holding power test results are shown in Table 3. The results suggest that the cohesion of formulation 2 with latent crosslinker DCP is similar to that of commercial benchmark Nitto 937UL, SG-03US, and formulation 0.

[070] Although the present invention has been described with reference to preferred embodiments, persons skilled in the art will recognize that changes may be made in form and detail without departing from the spirit and scope of the invention. All references cited throughout the specification, including those in the background, are incorporated herein in their entirety. Those skilled in the art will recognize, or be able to ascertain, using no more than routine experimentation, many equivalents to specific embodiments of the invention described specifically herein. Such equivalents are intended to be encompassed in the scope of the following claims.
CLAIMS

What is claimed is:

1. An adhesive composition comprising:
   An organosilicon-based pressure sensitive adhesive;
   a first crosslinker; and
   a second latent crosslinker, wherein the second latent crosslinker has a higher initiation temperature than the first crosslinker.

2. The adhesive composition according to claim 1, wherein the organosilicon-based pressure sensitive adhesive comprises polydialkylsiloxane, polyalkylarylsiloxane, polydiarylsiloxane, polyalkylalkenylsiloxane, or combinations thereof, or copolymers thereof.

3. The adhesive composition according to claim 1, wherein the organosilicon-based pressure sensitive adhesive comprises a DT or MQ resin.

4. The adhesive composition according to claim 1, wherein the organosilicon-based pressure sensitive adhesive comprises a solvent, wherein the solvent is chloroform, methylene chloride, 1,2-dichloroethane, benzene, toluene, xylene, heptane, hexane, isoparaffins, petroleum ether, 2,6-dichlorotoluene, or combinations thereof.

5. The adhesive composition according to claim 1, wherein the first crosslinker is benzoyl peroxide, 2,4-dichlorobenzoyl peroxide, or combinations thereof.

6. The adhesive composition according to claim 1, wherein the second latent crosslinker is cumyl hydrogen peroxide (CHP), dicumyl peroxide (DCP), tert-butyl hydrogen peroxide (TBHP), ditert-butyl peroxide (DTBP), 2,5-
dimethyl-2,5-di(t-butylperoxy)hexane (DPBH), 2,5-Di(tert-butylperoxy)-2,5-dimethyl-3-hexyne (LQ-DYBP), tert-butyl peroxybenzoate (TBPB), or combinations thereof.

7. The adhesive composition according to claim 1, wherein the ΔΤ between the first crosslinker and the second latent crosslinker is greater than 60 °C.

8. The adhesive composition according to claim 7, wherein the ΔΤ between the first crosslinker and the second latent crosslinker is greater than 80 °C.

9. The adhesive composition according to claim 8, wherein the ΔΤ between the first crosslinker and the second latent crosslinker is greater than 100 °C.

10. An adhesive construct comprising:
    an organosilicon-based pressure sensitive adhesive;
    a first crosslinker;
    a second latent crosslinker; and
    a backing, wherein the second latent crosslinker has a higher initiated temperature than the first crosslinker.

11. The adhesive construct according to claim 10, wherein the pressure sensitive adhesive comprises polydialkylsiloxane, polyalkylarylsiloxane, polydiarylsiloxane, polyalkylalkenylsiloxane, or combinations thereof, or copolymers thereof.

12. The adhesive construct according to claim 10, wherein the organosilicon-based pressure sensitive adhesive comprises a DT or MQ resin.
13. The adhesive construct according to claim 10, wherein the organosilicon-based **pressure sensitive adhesive** comprises a solvent, wherein the solvent is chloroform, methylene chloride, 1,2-dichloroethane, benzene, toluene, xylene, heptane, hexane, isoparaffins, petroleum ether, 2,6-dichlorotoluene, or combinations thereof.

14. The adhesive construct according to claim 10, wherein the first crosslinker is benzoyl peroxide, 2,4-dichlorobenzoyl peroxide, or combinations thereof.

15. The adhesive construct according to claim 10, wherein the second latent crosslinker is cumyl hydrogen peroxide (CHP), dicumyl peroxide (DCP), tert-butyl hydrogen peroxide (TBHP), ditert-butyl peroxide (DTBP), 2,5-dimethyl-2,5-di(t-butylperoxy)hexane (DPBH), 2,5-Di(tert-butylperoxy)-2,5-dimethyl-3-hexyne (LQ-DYBP), tert-butyl peroxybenzoate (TBPB), or combinations thereof.

16. The adhesive composition according to claim 10, wherein the ΔΤ between the first crosslinker and the second latent crosslinker is greater than 60 °C.

17. The adhesive composition according to claim 16, wherein the ΔΤ between the first crosslinker and the second latent crosslinker is greater than 80 °C.

18. The adhesive composition according to claim 17, wherein the ΔΤ between the first crosslinker and the second latent crosslinker is greater than 100 °C.

19. The adhesive construct according to claim 10, wherein the backing is paper, cloth, glass cloth, fiber glass, aluminum, silicone rubber, silicone resin-
treated glass cloth, polyethylene terephthalate (PET), polytetrafluoroethylene (PTFE), polychlorotrifluoroethylene (PCTFE), polyimide (PI) film, aromatic polyamide paper, polyvinyl chloride (PVC), polypropylene (PP), polyethylene (PE), or combinations thereof.

20. A method to prepare an adhesive construct comprising:
   an organosilicon-based pressure sensitive adhesive;
   a first crosslinker;
   a second latent crosslinker; and
   a backing, wherein the second latent crosslinker has a higher initiated temperature than the first crosslinker comprising the steps of:
   a) diluting the organosilicon-based pressure sensitive adhesive in toluene to a solid content of 30-60% and mixing until homogenous;
   b) adding 0.5-5% of a first crosslinker and adding 0.5-5% of a second latent crosslinker directly or dissolved in toluene to the diluted organosilicon-based pressure sensitive adhesive
   c) placing a coating table in a hood and placing a primed backing substrate on top of the coating table with primed side facing up;
   d) measuring appropriate amount of the adhesive composition from step b) and preparing a four-sided coater;
   e) coating the primed side of the backing substrate with the adhesive composition to provide the adhesive construct;
   f) placing the adhered construct in a first oven which has been heated to 90 °C for 2 minutes and then transferring the adhered construct to a second oven which has been heated to 180 °C for 2 minutes and then removing and allowing the adhered construct to cool to provide the bonded product.

21. The method of claim 20, wherein the organosilicon-based pressure sensitive adhesive comprises polydialkylsiloxane, polyalkylarylsiloxane, polydiarylsiloxane, polyalkylalkenylsiloxane, or combinations thereof, or copolymers thereof.
22. The method of claim 20, wherein the organosilicon-based pressure sensitive adhesive comprises a DT or MQ resin.

23. The method of claim 20, wherein the first crosslinker is benzoyl peroxide, 2,4-dichlorobenzoyl peroxide or combinations thereof.

24. The method of claim 20, wherein the second latent crosslinker is cumyl hydrogen peroxide (CHP), dicumyl peroxide (DCP), tert-butyl hydrogen peroxide (TBHP), ditert-butyl peroxide (DTBP), 2,5-dimethyl-2,5-di(t-butylperoxy)hexane (DPBH), 2,5-Di(tert-butylperoxy)-2,5-dimethyl-3-hexyne (LQ-DYBP), tert-butyl peroxynbenzoate (TBPB), or combinations thereof.

25. The adhesive composition according to claim 20, wherein the ΔT between the first crosslinker and the second latent crosslinker is greater than 60 °C.

26. The adhesive composition according to claim 25, wherein the ΔT between the first crosslinker and the second latent crosslinker is greater than 80 °C.

27. The adhesive composition according to claim 26, wherein the ΔT between the first crosslinker and the second latent crosslinker is greater than 100 °C.

28. The method of claim 20, wherein the backing is paper, cloth, glass cloth, fiber glass, aluminum, silicone rubber, silicone resin-treated glass cloth, polyethylene terephthalate (PET), polytetrafluoroethylene (PTFE), polychlorotrifluoroethylene (PCTFE), polyimide (PI) film, aromatic polyamide paper, polyvinyl chloride (PVC), polypropylene (PP), polyethylene (PE), or combinations thereof.
FIG. 1

FIG. 2
FIG. 3
**INTERNATIONAL SEARCH REPORT**

**A. CLASSIFICATION OF SUBJECT MATTER**

C09J 183/00(2006.01)i, C09J 183/04(2006.01)i

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)
C09J 183/00; C09J 183/06; C09J 179/08; C09J 183/04; C09J 7/02; C09K 5/54; C09J 183/10

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
Japanese utility models and applications for utility models

Electronic database consulted during the international search (name of database and, where practicable, search terms used)
ekOMPASS(KIPO internal) & Keywords: organosiloxane, adhesive, PSA, benzoyl peroxide, ¼, ½, ¾, ½, 가교재, 과산화물, 오르기노, 산복신, crosslinker, hydroperoxide, DT resin, MQ resin, cumen hydroperoxide

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

<table>
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<tr>
<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
<th>Relevant to claim No.</th>
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<td>JP 4809988 B2 (NITTO DENT CO CORPORATION) 09 November 2011; and claims 1-6.</td>
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* Further documents are listed in the continuation of Box C.  
** See patent family annex.

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier application or patent but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as indicated)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

Date of the actual completion of the international search: 03 March 2016 (03.03.2016)

Date of mailing of the international search report: 03 March 2016 (03.03.2016)

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