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(54) Title: ADHESIVE ACTIVATION BY A SKIN EFFECT OF AN ALTERNATE CURRENT AT LOW FREQUENCIES

(57) Abstract: A process for the activation of a heat activated material comprising heating the material by a combination of the Joule effect and the skin effect whereby a piece of the material is located adjacent to a metal foil that comprises part of an alternating current circuit whereby the material is activated by heat generated by the flow of current through the metal foil both by the Joule effect and the skin effect, the invention may be used in a variety of industries such as the transportation, construction and furniture industries.

ADHESIVE ACTIVATION BY A SKIN EFFECT OF AN ALTERNATE CURRENT AT LOW FREQUENCIES

The present invention relates to process for the activation of adhesives and is particularly suitable for rapid activation in an environment in which activation is difficult to achieve by baking in an oven or heating in a press or an autoclave.

The invention further provides a new and improved way to activate bonding and/or expanding material and may be used for the activation of a foamable adhesive material to cause both foaming and curing together with the development of adhesive properties.

The invention is particularly useful in a variety of industries such as the transportation industries including automotive, aerospace and rail industries and the construction and furniture industries. One particular use is in the repair of adhesive bonds which may have been damaged in, for example, an automobile crash.

By activation is meant to trigger a physical and/or chemical reaction within an adhesive formulation. For example, an adhesive may contain a thermo-curable material perhaps in combination with a material that can be activated by heat to cause the material to cure. In this instance activation comprises the initiation of the activities that cause the thermo-curable material to cure. In many instances curing may involve crosslinking. Similarly, an adhesive may be thermo-foamable either as an alternative to being thermo-curable or in addition to being thermo-curable. In this instance activation causes the adhesive to foam. Where the adhesive is both thermo-foamable and thermo-curable activation can include both foaming and curing and the activation temperature for the initiation of foaming may be the same or different than for the activation temperature for curing. However, in relation to a thermo-foamable material which may contain physical and/or chemical blowing agents that produce gasses to cause foaming activation comprises the initiation of the activities that cause the material to foam.

Adhesive are frequently based on polymeric materials whose composition can be changed by changing certain circumstances that result in the development of adhesive properties. Adhesives may be activated at ambient temperature through the use of certain catalysts. In addition they may be activated by heat such as by passage through an oven, in a hot press or in an autoclave. In the transportation, construction and furniture industries thermo-curable adhesives which may also be thermo-foamable are frequently used for providing structural reinforcement of increased strength at bond points. Thermo-foamable adhesive are also used to produce sound insulation and the like and in this instance the soft foam typically

used in acoustic baffles may be required to develop adhesive properties as it foams. In these industries the heat required for curing and/or foaming is generally derived from an oven, a hot press or an autoclave.

There are other types of activation such as the use of ultra violet light, the reaction to moisture, the projection of IR light or microwave.

DE 102009031419 suggests a process for producing heat by controlling the skin effect which employs a metal foil driven by an alternating current at frequencies between 20-60 kHz.

It is known to produce heat in electrical conducting elements by what is known as the skin effect which is the tendency for alternating electric current to distribute itself within a conductor with the current density being largest near the surface of the conductor. The Joule effect on the other hand is the heat generated by the current flowing through a conductor.

The present invention concerns the activation of the material without using convection heating in an oven or an autoclave. The invention is aimed at providing rapid activation of the adhesive so that the desired adhesion can be achieved rapidly which is particularly useful during normal assembly line operations and, in some instances, enables assembly line operations to be speeded up.

The present invention therefore provides a process for the activation of a heat activated material comprising heating the material by means of a combination of the Joule effect and the skin effect whereby the material is located adjacent to a metal foil that comprises part of an alternating current circuit whereby the material is activated by heat generated by the flow of current through the metal foil both by the Joule effect of induction and the skin effect. In a preferred embodiment the heat activated material is a heat activated adhesive such as a heat curable adhesive and is preferably heat curable and heat foamable. In this instance the material may be both foamed and cured by the heat generated by the flow of current through the metal foil. The use of the combination of heating results in rapid development of the desired temperature. This means that the activation can be readily integrated into assembly lines and other industrial processes and in certain instances the speed of the assembly line can be increased.

The activatable material which may be based on a thermosetting or thermoplastic polymer is located close to the metallic foil and may be sandwiched between the “foil” (or at least one

foil) and a substrate such as a metal substrate to which it is to be bonded. The structure can be spaced by voids such as air to allow for expansion of the layers of the activatable material. The size and shape of the piece of activatable material will depend on the cavity in which it is to be employed. However, the foil of the heat generating circuit preferably extends over the full area of the piece of material. The foil is then connected to an AC generator, which preferably runs from one end to another of the foil.

The intensity and frequency of the current passing through the foil results in a fast motion of electrons in the metal foil, this displacement causes heating of the foil by the Joule effect so that the heat dissipated by the foil can be used to activate the material. In addition, the alternating electric current (AC) has a tendency to distribute itself near the surface of the foil. This is known as the skin effect. The skin effect creates heat at the surface of the foil adjacent to the activatable material to allow rapid heating of the activatable material. Additionally the skin effect may have the advantage that it can localise the heat and hence avoid possible damage which may occur if it is required to heat the whole foil, to cause activation of the material.

The activatable adhesive material may be foamable or non-foamable and preferably includes heat conductive fillers such as Iron Powder, Nitrile Boron, Aluminium Nitride, Silicon Carbide, Boron Carbide, the Ferrophos or carbon fibres. The presence of such fillers enhances and accelerates the heating process and from 3 wt %, preferably 5 wt % to 75 wt % of such fillers are preferred.

The preferred choice of foil, the thickness of the foil and the frequency of the alternating current will depend on the heat required for activation of the heat activatable material. However, we have found that for the activation of adhesives such as those used in the transportation, construction and furniture industries, particularly thermo foamable and thermo hardenable adhesive the use of a conductive metal foil particularly of stainless steel, copper or aluminium of thickness from 0.05 to 2 mm preferably 0.05 to 0.2 mm with a current of from 10 kHz to 1MHz preferably 20 kHz to 500kHz more preferably 100 kHz to 400 kHz is particularly useful.

When the activatable material is a foamable material it will be selected according to the properties the foam is required to provide wherever it is to be located. The invention is particularly useful when the foam is required to provide sealing and/or sound absorption in which case a high expansion, typically 100%-3000% preferably 400-2500% expansion foamable material may be used to produce a soft foam. Alternatively the foam may be

required to reduce vibration and/or hardness in a vehicle and/or it may be required to provide crash resistance. If the foam is to provide a reinforcing effect it may be a rigid foam provided from a foamable material with a relatively low degree of expansion, typically 30% to 200% and it may also be a material that cross-links at a similar temperature to that at which it foams. A cross-linkable epoxy resin based formulation may be used. Preferred foamable materials are formulations that are thermoplastic at temperatures below that at which they foam and, accordingly can be extruded or injection moulded to provide the foamable material at temperatures below the foaming temperature. Upon heating to cause foaming the material may be formulated so that it will cross-link if a rigid reinforcing foam is required. The foamable material preferably includes a tackifier such as a petroleum resin or a rosin ester to aid the adhesion of the material upon foaming.

The invention is particularly useful in the provision of sound insulation within automobile cavities when the foamable material is required to provide sound insulation and/or sealing the material may have a degree of expansion such as from 200% to 3000%. It may also be a soft foam without cross linking.

In the preferred production of sound insulation for automobiles the material is foamable and is such as to produce a highly expanded soft foam typically from polymers such as ethylene unsaturated ester copolymers typically ethylene vinyl acetate copolymers and/or ethylene acrylate copolymers. In this embodiment a particularly preferred material is an olefinic polymer-based acoustic foam, and more particularly an ethylene based polymer. For example, the foamable material may be based on an ethylene copolymer or terpolymer that may contain a C_3 to C_8 alpha-olefin comonomer. Examples of particularly preferred polymers include ethylene vinyl acetate copolymers, ethylene acrylate copolymers, EPDM, or mixtures thereof. Other examples of preferred foam formulations that are commercially available include polymer-based materials commercially available from L & L Products, Europe, under the designations as L-2704, L-2806, L-2811, L-2820, L-2821, L-1066, L-2105, L-2106, L-2115, L-2308, L-2411, L-2412, L-2663, L-2664, L-2700, L-2703, L-4161, L-4200, L-4300, L-4315, L-4316, L-7102, L-7107 and L-7220.

A number of other suitable materials are known in the art and may also be used for producing foams for noise attenuation and/or vibration damping. One such foam includes an open-cell polymeric base material, such as an ethylene-based polymer which, when compounded with appropriate ingredients (typically a blowing and curing agent), expands and cures in a reliable and predictable manner upon the application of heat or the occurrence of a particular ambient condition. From a chemical standpoint for a thermally

activated material, an acoustic foam is usually initially processed as a flowable thermoplastic material before curing. It will preferably cross-link upon curing, which makes the material resistant to further flow or change of final shape.

While the preferred materials for fabricating a sound absorption and/or vibration damping material have been disclosed, the material can be formed of other materials (e.g., foams regarded in the art as structural foams) provided that the material selected is heat-activated or otherwise activated by an ambient condition (e.g. moisture, pressure, time or the like) and cures in a predictable and reliable manner under appropriate conditions for the selected application.

Some other possible materials include, but are not limited to, polyolefin materials, copolymers and terpolymers, phenol/formaldehyde materials, phenoxy materials, and polyurethanes. US Patent Nos. 5,266,133; 5,766,719; 5,755,486; 5,575,526; 5,932,680; and WO00/27920 describe suitable materials. In general, the desired characteristics of the resulting foam include relatively low glass transition point, and good corrosion resistance properties. In this manner, the material does not generally interfere with the materials systems employed by automobile manufacturers. Moreover, it will withstand the processing conditions typically encountered in the manufacture of a vehicle, such as the e-coat priming, cleaning and degreasing and other coating processes.

Generally, suitable expandable foams have a range of expansion ranging from approximately 30 to over 1000 percent. The level of expansion of the acoustical foam may be to as high as 1500 to 2000 percent or more.

In another embodiment, a sound absorption material may be provided in an encapsulated or partially encapsulated form, for instance an expandable foamable material is encapsulated or partially encapsulated in an adhesive shell. Moreover, the sound absorption material may include a melt-flowable material such as that disclosed in US Patent No. 6,030,701.

One or more curing agents may be included in the activatable material used in this invention. Optionally curing agent accelerators may also be included. The amounts of curing agents and curing agent accelerators used can vary widely depending upon the type of structure desired, the desired properties of the material and the desired amount of any expansion of the foamable material and the desired rate of expansion. Exemplary ranges for the curing agents or curing agent accelerators present in the foamable material range from about 0.001% by weight to about 7% by weight.

Preferably, the curing agents assist in curing by cross linking of the polymers, phenoxy epoxy resins or both and any epoxy resin that may be present. It is also preferable for the curing agents to assist in thermosetting the material. Useful classes of curing agents are materials selected from aliphatic or aromatic amines or their respective adducts, amidoamines, polyamides, cycloaliphatic amines, anhydrides, polycarboxylic polyesters, isocyanates, phenol-based resins (e.g., phenol or cresol novolak resins, copolymers such as those of phenol terpene, polyvinyl phenol, or bisphenol-A formaldehyde copolymers, bishydroxyphenyl alkanes or the like), or mixtures thereof. Particular preferred curing agents include modified and unmodified polyamines or polyamides such as triethylenetetramine, diethylenetriamine tetraethylenepentamine, cyanoguanidine, dicyandiamides and the like. If an accelerator for the curing agent is used examples of materials includes a modified or unmodified urea such as methylene diphenyl bis urea, an imidazole or a combination thereof.

Depending upon the function required of the material, it may include one or more additional polymers or copolymers, which can include a variety of different polymers, such as thermoplastics, elastomers, plastomers and combinations thereof. For example, and without limitation, polymers that might be appropriately incorporated into the material include halogenated polymers, polycarbonates, polyketones, polyurethanes, polyesters, and polymers derived from silanes, sulfones, allyls, olefins, styrenes, acrylates, methacrylates, epoxies, silicones, phenolics, rubbers, polyphenylene oxides, terphthalates, acetates (e.g., EVA), acrylates, methacrylates (e.g., ethylene methyl acrylate polymer) or mixtures thereof. Other potential polymeric materials may be or may include, without limitation, polyolefin (e.g., polyethylene, polypropylene) polystyrene, polyacrylate, poly(ethylene oxide), poly(ethyleneimine), polyester, polyurethane, polysiloxane, polyether, polyphosphazine, polyamide, polyimide, polyisobutylene, polyacrylonitrile, poly(vinyl chloride), poly(methyl methacrylate), poly(vinyl acetate), poly(vinylidene chloride), polytetrafluoroethylene, polyisoprene, polyacrylamide, polyacrylic acid, polymethacrylate.

When used, these polymers can comprise a small portion or a more substantial portion of the material. When used, the one or more additional polymers preferably comprises about 0.1% to about 50%, more preferably about 1% to about 20% and even more preferably about 2% to about 10% by weight of the foamable material.

Examples of suitable rigid materials include epoxy-base resins and examples of such foamable materials are the products L-0507, L-5207, L-5214, L-5234, L-5235, L-5236, L-

5244, L-8050 and L-8150, which are commercially available from L & L Europe, Strasbourg, France. The product should be chosen so that it can be shaped at temperatures below that at which it will be activated, typically 80°C to 90°C. It is further preferred that where it is used in automobile frame or seat manufacture it expands at the temperatures experienced in the oven used to dry and cure the anticorrosion coating deposited in the e-coat process, typically 120°C to 180°C, more typically 130°C to 150°C. Prior to activation, the material is preferably dry and not tacky to the touch, since this facilitates shipping and handling and prevents contamination.

Epoxy resin is used herein to mean any of the conventional dimeric, oligomeric or polymeric epoxy materials containing at least one epoxy functional group. The epoxy content is typically more than 40%. Moreover, the term epoxy resin can be used to denote one epoxy resin or a combination of multiple epoxy resins. The polymer-based materials may be epoxy-containing materials having one or more oxirane rings polymerizable by a ring opening reaction. In a preferred embodiment, the material includes between about 2% and 75% by weight epoxy resin, more preferably between about 4% and 60% by weight epoxy resin and even more preferably between about 25% and 50% by weight epoxy resin. Of course, amounts of epoxy resin may be greater or lower depending upon the intended application of the activatable material.

The epoxy may be aliphatic, cycloaliphatic, aromatic or the like. The epoxy may be supplied as a solid (e.g., as pellets, chunks, pieces or the like) or a liquid (e.g., an epoxy resin) although liquid resins are preferred to enhance process ability of the adhesive formulation. As used herein, unless otherwise stated, a resin is a solid resin if it is solid at a temperature of 23°C and is a liquid resin if it is a liquid at 23°C. The epoxy may include an ethylene copolymer or terpolymer.

An epoxy resin may be added to increase the adhesion, flow properties or both of the material which may be foamable or unfoamable. One exemplary epoxy resin may be a phenolic resin, which may be a novolac type or other type resin. Other preferred epoxy containing materials may include a bisphenol-A epichlorohydrin ether polymer, or a bisphenol-A epoxy resin which may be modified with butadiene or another polymeric additive or bisphenol-F-type epoxy resins. Moreover, various mixtures of several different epoxy resins may be employed as well. Examples of suitable epoxy resins are sold under the tradename Araldite GY 282, GY 281 and GY 285 supplied by Huntsman.

In certain embodiments, it may be desirable to include one or more thermoplastic polyethers and/or thermoplastic epoxy resins in the activatable material which may or may not be foamable. When included, the one or more thermoplastic polyethers preferably comprise between about 1% and about 90% by weight of the material, more preferably between about 3% and about 60% by weight of the material and even more preferably between about 4% and about 25% by weight of the material. As with the other materials, however, more or less thermoplastic polyether may be employed depending upon the intended use of the material.

The thermoplastic polyethers typically include pendant hydroxyl moieties. The thermoplastic polyethers may also include aromatic ether/amine repeating units in their backbones. The thermoplastic polyethers of the present invention preferably have a melt index between about 5 and about 100, more preferably between about 25 and about 75 and even more preferably between about 40 and about 60 grams per 10 minutes for samples weighing 2.16 Kg at a temperature of about 190°C. Of course, the thermoplastic polyethers may have higher or lower melt indices depending upon their intended application. Preferred thermoplastic polyethers include, without limitation, polyetheramines, poly(amino ethers), copolymers of monoethanolamine and diglycidyl ether, combinations thereof or the like. Preferably, the thermoplastic polyethers are formed by reacting an amine with an average functionality of 2 or less (e.g., a difunctional amine) with a glycidyl ether (e.g., a diglycidyl ether). As used herein, the term difunctional amine refers to an amine with an average of two reactive groups (e.g., reactive hydrogens).

According to one embodiment, the thermoplastic polyether is formed by reacting a primary amine, a bis(secondary) diamine, a cyclic diamine, a combination thereof or the like (e.g., monoethanolamine) with a diglycidyl ether or by reacting an amine with an epoxy-functionalized poly(alkylene oxide) to form a poly(amino ether). According to another embodiment, the thermoplastic polyether is prepared by reacting a difunctional amine with a diglycidyl ether or diepoxy-functionalized poly (alkylene oxide) under conditions sufficient to cause the amine moieties to react with the epoxy moieties to form a polymer backbone having amine linkages, ether linkages and pendant hydroxyl moieties. Optionally, the polymer may be treated with a monofunctional nucleophile which may or may not be a primary or secondary amine.

Additionally, it is contemplated that amines (e.g., cyclic amines) with one reactive group (e.g., one reactive hydrogen) may be employed for forming the thermoplastic polyether. Advantageously, such amines may assist in controlling the molecular weight of the thermoplastic ether formed.

Examples of preferred thermoplastic polyethers and their methods of formation are disclosed in US Patents Nos. 5,275,853; 5,464,924 and 5,962,093. Advantageously, the thermoplastic polyethers can provide the material with various desirable characteristics such as desirable physical and chemical properties for a wide variety of applications as is further described herein.

Although not required, the formulation may include one or more ethylene polymers or copolymers such as ethylene acrylate, copolymers and ethylene acetate copolymers. Ethylene methacrylate and ethylene vinyl acetate are two preferred ethylene copolymers.

It may also be desirable to include a reactive polyethylene resin that is modified with one or more reactive groups such as glycidyl methacrylate or maleic anhydride. Examples of such polyethylene resins are sold under the tradename LOTADER® (e.g., LOTADER AX 8900) and are commercially available from Arkema Group.

When the activatable material is foamable one or more blowing agents may used to cause the material to be foamable by producing inert gasses that form, as desired, an open and/or closed cellular structure of the foamed material.

The blowing agent may include one or more nitrogen containing groups such as amides, amines and the like. Examples of suitable blowing agents include azodicarbonamide, dinitrosopentamethylenetetramine, azodicarbonamide, initrosopentamethylenetetramine, 4,4$_i$-oxy-bis-(benzenesulphonylhydrazide), trihydrazinotriazine and N, N$_i$-dimethyl-N,N$_i$-dinitrosoterephthalamide. An accelerator for the blowing agents may also be provided. Various accelerators may be used to increase the rate at which the blowing agents form inert gasses. One preferred blowing agent accelerator is a metal salt, such as an oxide, for example zinc oxide. Other preferred accelerators include modified and unmodified thiazoles or imidazoles. The amounts of blowing agents and blowing agent accelerators that should be used can vary widely depending upon the type of cellular structure desired, the desired amount of expansion the desired rate of expansion and the like. Exemplary ranges for the amounts of blowing agents and blowing agent accelerators in the activatable material range from about 0.001% by weight to about 5% by weight.

The activatable material may also include one or more fillers, including but not limited to particulate materials (e.g., powder), beads, microspheres such as Zeospheres available from Zeelan Industries, or the like. Preferably the filler includes a material that is generally non-

reactive with the other components present in the activatable material. While the fillers may generally be present to take up space at a relatively low weight, it is contemplated that the fillers may also impart properties such as strength and impact resistance.

Examples of fillers that may be used include silica, diatomaceous earth, glass, clay (e.g., including nanoclay), talc, pigments, colorants, glass beads or bubbles, glass, carbon or ceramic fibers, nylon or polyamide fibers (e.g., Kevlar), antioxidants, and the like. Such fillers, particularly clays, can assist in leveling itself during flow of the foamable material. The clays that may be used as fillers may include clays from the kaolinite, illite, chloritem, smecitite or sepiolite groups, which may be calcined. Examples of suitable fillers include, without limitation, talc, vermiculite, pyrophyllite, sauconite, saponite, nontronite, montmorillonite or mixtures thereof. The clays may also include minor amounts of other ingredients such as carbonates, feldspars, micas and quartz. The fillers may also include ammonium chlorides such as dimethyl ammonium chloride and dimethyl benzyl ammonium chloride. Titanium dioxide might also be employed.

In one preferred embodiment, one or more mineral or stone type fillers such as calcium carbonate, sodium carbonate or the like may be used as fillers. In another preferred embodiment, silicate minerals such as mica may be used as fillers.

When employed, the fillers can range from 10% or less to 90% or greater by weight of the material, but more typical from about 20 to 55% by weight of the material. According to some embodiments, the material may include from about 0% to about 3% by weight, and more preferably slightly less that 1% by weight clays or similar fillers. Powdered (e.g. about 0.01 to about 50, and more preferably about 1 to 25 micron mean particle diameter) mineral type filler can comprise between about 5% and 70% by weight, more preferably about 10% to about 50% by weight.

Other additives, agents or performance modifiers may be included in the material as desired, including but not limited to an antioxidant, a UV resistant agent, a flame retardant, an impact modifier, a heat stabilizer, a colorant, a processing aid, a lubricant, a reinforcement (e.g., chopped or continuous glass, ceramic, aramid, or carbon fiber, particulates or the like). Liquid polysufides may be used to improve the environmental exposure such as exposure to humidity and salt water.

In a preferred embodiment the activatable material contains an adhesion promoting material such as a petroleum resin or a synthetic or naturally occurring rosin ester tackifier. The use

of these materials can improve the adhesion of the material to the foil and can also improve the adhesion of the material to the walls of a cavity. It is preferred to include from 0.5 to 10 wt % of such an adhesion promoting material

When determining appropriate components for the activatable material, it may be important to formulate the material so that it will be activated which may include foaming at appropriate times or temperatures. For instance, in some applications, it is undesirable for the material to be reactive at room temperature or otherwise at the ambient temperature in a production environment. More typically, the material flows at higher processing temperatures. As an example, temperatures such as those encountered in an automobile assembly plant may be appropriate, especially when the material is processed along with the other components at elevated temperatures or at higher applied energy levels, e.g., during painting preparation steps. In addition it is preferable that the material be non-tacky to the touch at ambient temperature to reduce the pickup of dirt and dust and to facilitate storage and transportation.

The amount of material employed will be determined by the size of the cavity in which the foam is to be provided and the desired degree of expansion. However for most vehicles a laminar structure containing a layer of foamable material from 0.3 mm to 10 mm thick particularly 1.5 mm to 5 mm has been found to be appropriate particularly for the production of a sound absorption and/or vibration damping foam such a thickness is often required in vehicle repair activities.

In a further embodiment the present invention may be used for the structural reinforcement of cavities in body frames and in seats of automobiles or other transportation systems such as trucks, buses, railroad vehicles and aircraft. Here the material is generally a rigid reinforcing thermosetting layer such as a foamable epoxy resin or a foamable polyurethane which may be a blocked polyurethane. In this instance the material is foamable and serves two main functions, it will expand across the space between the foil and the interior of the hollow section and will bond to some or all of the interior walls of the hollow section. Activation therefore enables the foamable material to expand and fill a gap between the foil and a hollow structure it is designed to reinforce and to bond to selected internal surfaces of the hollow structure. Accordingly the foamable material must expand at the desired temperature and be sufficiently adhesive to firmly bond the reinforcing member inside the vehicle structure. In this embodiment once foamed it should be sufficiently strong that it does not contribute any weakness to the overall reinforcing effect provided.

The cross section of the foil employed in the alternating current circuit to provide the heat in the present invention is preferably constant in order to generate a constant heat supply to the adhesive material and the foil is preferably of thickness 0.05 to 2 mm more preferably 0.05 to 0.25 mm, most preferably 0.05 to 0.15 mm. The foil may be of any suitable electrically conductive material; stainless steel, copper and aluminium being preferred. The frequency of the alternating current is also preferably constant, and will depend upon the activation temperature of the adhesive and the dimension of the metal foil. This temperature is regulated to be constant. Currents having frequencies in the range 10kHz to 1MHz are preferred, more preferably 100kHz to 400kHz. The invention is particularly useful for the activation of preformed pieces of activatable adhesive material which may be injection moulded, extruded or die cut. The invention is useful for the activation of adhesives which may or may not be foamable. It is however especially useful for the activation of thermofoamable and thermocurable preformed adhesive materials of thickness from 0.3 to 10 mm particularly from 1.5 to 5 mm.

The present invention is illustrated by reference to the accompany figures in which

Figure 1 is a schematic diagram showing an alternating current electric circuit (1) contacting a metal foil (2) and a strip of expandable material (3) located adjacent to the foil.

Figure 2 shows how the strip may be located close to but spaced apart from the metal foil and Figure 3 shows how the strip may be touching the metal foil;

Figure 4 and 5 show the materials of Figures 2 and 3 after expansion of the foamable material.

The invention is further illustrated by reference to the following examples which employed an apparatus similar to that shown in Figure 1 the metal foil was a stainless steel strip of a thickness of 0.1mm. The expandable material is a strip of the ethylene vinyl acetate copolymer based material available from L&L Products as L-4161 to which had been added 5 wt % iron powder (except for Example 4). The strip was of thickness 2.5 mm prior to expansion.

The expandable material was located on a metal strip and it was activated by passage of a current of 58A so that the surface of the metal strip reached the required peak temperature and then with 25A to maintain the temperature. The power was 11V and the frequency was 20kHz through the metal foil which caused the expandable material to foam and develop

adhesive properties so as to bond to the metal strip. The laminate formed of the expanded material and the metal strip was allowed to cool and stand for a certain period of time and the strength of the bond (the lap shear) between the metal strip and the expanded material was measured. The degree of expansion of the expandable material was about 150%. The results were as follows.

Examples	Iron Powder Filler	Heating time (seconds)	Surface Temperature of foil °C	Stand time	Lap Shear (Newtons)
1	Yes	90	160	24 hours	300
2	Yes	90	140	24 hours	300
3	Yes	90	150	20 minutes	260
4	No	90	150	20 minutes	150
5	Yes	90	150	3 days	375

The results of Examples 1 and 2 show that the temperature of the surface of the metal foil has little effect on the adhesive strength if it is above the activation temperature of the material.

The results of Examples 1, 2, 3 and 5 show that the stand time has an influence on the adhesive strength. The longer is the stand time, the stronger is the adhesion as the material continues to cure after heating is completed.

The results of the tests on Examples 3 and 4 show that the presence of thermal conductive fillers such as iron powder in the adhesive material increase the adhesive strength.

The present invention therefore provides a convenient method for the activation of adhesives which is particularly useful in the transportation industries such as automobile, aerospace, and rail industries as well as the construction and furniture industries. It is particularly useful in the repair of damaged areas of automobiles, aerospace and rail vehicles especially in confined spaces. Advantages of this method are:

- Fast activation of the material
- Activation with lightweight tools of a low volume
- The ability to activate a material without the need to pass through an oven, a hot press or an autoclave

- Very localised heat activation which avoids damage to other non-heat resisting parts that are nearby
- Cost efficient process (compared to an autoclave, an oven etc.)

A further advantage of the invention is that the part of the activatable material does not need to be very close to the heating tool (the metallic foil) to be activated. Furthermore, the environment can be metallic provided there is electrical insulation between the metal foil through which the current is passing and the environment itself and the insulation may be made by adhesive. This is particularly useful in repair situations.

CLAIMS

1. A process for the activation of a heat activated material comprising heating the material by a combination of the Joule effect and the skin effect whereby a piece of the material is located adjacent to a metal foil that comprises part of an alternating current circuit whereby the material is activated by heat generated by the flow of current through the metal foil both by the Joule effect and the skin effect.

2. A process according to Claim 1 in which the heat activated material is a heat activated adhesive.

3. A process according to Claim 1 or Claim 2 in which the heat activated material is heat curable and heat foamable.

4. A process according to any of the preceding claims in which the activatable material is a thermosetting or thermoplastic resin.

5. A process according to any of the preceding claims in which the foil extends over the full area of the piece of material.

6. A process according to any of the preceding claims in which the activatable material includes heat conductive fillers.

7. A process according to Claim 6 in which the fillers are selected from Nitrile Boron, Aluminium Nitride, Silicon Carbide, Boron Carbide, the Ferrophos or carbon fibres.

8. A process according to any of the preceding claims in which the conductive metal foil is of stainless steel, copper or aluminium and of thickness from 0.05 to 2 mm preferably 0,.05 to 0.2 mm with a current of from 10 kHz to 1 MHz preferably 20 kHz to 500 kHz more preferably 100 kHz to 400 kHz.

9. A process according to any of the preceding claims for the provision of sound insulation within automobile cavities.

10. A process according to any of the preceding claims in which the activatable material is a foamable epoxy-base resin.

11. A process according to any of the preceding claims in which the activatable material comprises a layer of foamable material from 1.5 mm to 10 mm thick, particularly 1.5 mm to 5 mm.

12. A process according to any of the preceding claims in which the foil is of thickness 0.05 to 2 mm.

13. A process according to any of the preceding claims in which the frequency of the alternating current is constant.

14. A process according to any of the preceding claims in which the current has a frequency of 10 kHz to 1 MHz.

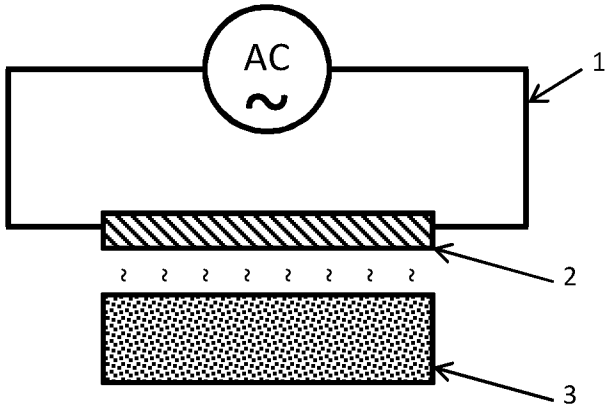

FIGURE 1

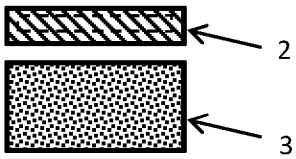

FIGURE 2

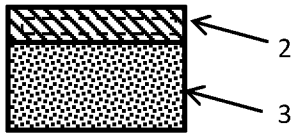

FIGURE 3

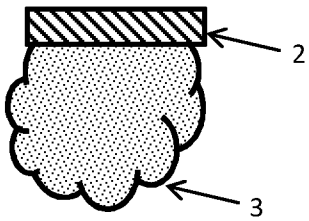

FIGURE 4

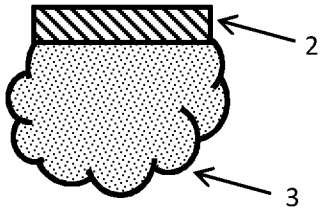

FIGURE 5

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2012/056232

A. CLASSIFICATION OF SUBJECT MATTER
INV. C09J5/00
ADD.

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

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Y	the whole document paragraphs [0010], [0011], [0018]; claims 1,3,4	1-14

Y	US 2007/128367 A1 (TABAKOVIC RIFAT [US] ET AL) 7 June 2007 (2007-06-07) the whole document	1-14

Y	US 2004/131839 A1 (EAGLE GLENN G [US]) 8 July 2004 (2004-07-08) the whole document	1-14

Y	JP 2004 331858 A (HORI GLASS CO LTD) 25 November 2004 (2004-11-25) abstract	1-14

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents :

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"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

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
31 August 2012	07/09/2012

Name and mailing address of the ISA/	Authorized officer
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INTERNATIONAL SEARCH REPORT

Information on patent family members

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