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(54) VIBRATION DAMPING CONSTRUCTIONS USING AROMATIC EPOXY HIGH TEMPERATURE DAMPING MATERIALS

VIBRATIONDAEMPFENDE KONSTRUKTIONEN UNTER VERWENDUNG VON AROMATISCHEN EPOXIDMATERIALIEN, DIE BEI HOHEN TEMPERATUREN DAEMPFEN

STRUCTURES AMORTISSANT LES VIBRATIONS FAISANT APPEL A DES MATERIAUX AMORTISSANTS A HAUTE TEMPERATURE EN EPOXY AROMATIQUE

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• **SCIENTIFIC AMERICAN** vol. 220, January 1969, **NEW YORK US** pages 98 - 106 T.P. PIN 'The Control of Vibration and Noise'
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Description

Background Of The Invention

5 1. Field of the Invention

This invention relates to vibration-damping constructions and a method useful for damping vibratory and/or noise emitting structures and component parts of devices such as automobiles, aircraft, industrial equipment, and appliances. This vibration-damping construction comprising at least a single layer of aromatic epoxy high temperature damping materials.

10 2. Description of Related Art

As technology moves toward energy conservation with the concomitant drive towards light weight structures that move at faster speeds and operate at higher temperatures, the acoustic and vibratory responses become larger and less desirable.

It has long been known that the vibration of component parts of devices and structures that vibrate under the influence of an applied internal or external force can be substantially reduced by the attachment of a layer of viscoelastic material. For example, U.S. Patent No. 3,640,836 discloses a vibration-damping laminate in which the viscoelastic layer is a polymer comprised of ethylene, vinyl acetate and acrylic and/or methacrylic acid. U.S. Patent No. 3,847,726 discloses a viscoelastic adhesive composition of a polyepoxide, a polyether amine, a heterocyclic amine, and a phenol useful as vibration-damping material over a -25° to +60°C range. Such compositions, however, are not effective for vibration-damping over prolonged periods of time at elevated temperatures.

U.S. Patent No. 3,833,404 discloses viscoelastic damping compositions comprising an interpenetrating polymer network composition consisting essentially of 5-95% by weight of a polyalkyl acrylate elastomer, for example, polyethyl acrylate or polybutyl acrylate, polyvinyl acetate, polyurethane, polybutadiene, natural rubber, silicone rubber, butyl rubber, chloroprene, ethylenepropylene terpolymer elastomers, polyvinyl alcohol, thiol rubber, and copolymers thereof; and 95-5% by weight of a plastic, such as polystyrene, poly- α -methyl styrene, polyalkyl acrylates, for example, polymethyl methacrylate or polyethyl methacrylate, poly-1-alkenes, for example, polypropylene, polyacrylic acid, and copolymers thereof, together with an outer plastic constraining layer.

Epoxies have traditionally been used as constraining layers in damping constructions since they do not exhibit any significant damping due to their highly crosslinked networks. Although U.S. Patent No. 3,833,404 considers epoxy for the constraining layer, epoxy is not considered alone or in combination with other polymers as a potential viscoelastic damping material in this document, contrary to the previously cited US-A-3847726.

U.S. Patent No. 4,385,139 discloses a synthetic resin composition composed of at least two different polymers and a filler for use as starting material for vibration-damping sheets. In addition to at least one acrylic acid ester, at least one vinyl ester and inorganic filler, 1 to 10% by weight of an epoxide resin is added to the mixture relative to the sum total of the first 3 components. Epoxy resins with fluorenes are not disclosed in this document.

U.S. Patent No. 4,304,694 discloses a damping composite comprising a resin matrix of about 20 to 43% by weight of a flexible epoxy, about 12 to 35% by weight of a stiff epoxy, about 35 to 61% by weight of a flexible crosslinking agent and about 20 to 50% by weight of a high modulus graphite fiber. The crosslinking agent described comprises a long chain amine-fatty acid amide.

U.S. Patent No. 4,447,493 discloses a constrained-layer damping construction containing a viscoelastic polymer that is the reaction product of (a) 25 to 75 weight percent of an acryloyl or methacryloyl derivative of at least one oligomer having a T_g of less than 25°C and a molecular weight per oligomer of 600 to 20 000 and (b) 75 weight percent of a monomer whose homopolymer has a T_g of at least 50°C, the copolymer being suitable for damping vibrations at relatively high temperature, for example, 50° to 150°C. It appears that the 50°-150°C damping regime was measured at a frequency of 1000 Hz. Since damping temperatures generally decrease about 6 to 7°C with every decreasing decade of frequency, the copolymers described in the '493 patent would be expected to damp between about 30° and 130°C at 1 Hz.

U.S. Patent No. 4,684,678 discloses epoxy resin compositions that on curing yield cured resins having a high glass transition temperature, high ductility, and low moisture pick-up. 9,9-bis(aminophenyl)fluorenes are used as the curing agents. It discloses some of the fluorene containing epoxy resins of the present invention which have glass transition temperatures in the range presently claimed in claim 2.

U.S. Patent No. 4,707,534 discloses diglycidyl ethers of ortho-substituted-4-hydroxyphenylfluorenes curable compositions comprising diglycidyl ethers, and cured resins thereof. The cured resins have a high glass transition temperature and improved modulus of elasticity.

Summary Of The Disclosure

Briefly, in one aspect of the present invention a method of using cured fluorene-containing epoxy resins as vibration-damping materials is provided. The method for damping the vibration of a vibrating solid article at temperatures above 120°C to approximately 275°C at 1 Hz comprises providing a viscoelastic layer construction with at least one layer of a cured fluorene-containing epoxy resin.

In another aspect of the present invention, a damping construction is provided, wherein the damping construction comprises at least one layer of viscoelastic material applied to a vibratory solid article, such that the viscoelastic material comprises a cured fluorene-containing epoxy resin.

In one variation of the damping construction of the present invention, the cured fluorene-containing epoxy resin is bonded to a vibratory solid article that is to be damped. This application is referred to as a "free" viscoelastic layer, sometimes referred to as "extensional" damping. See Kerwin and Ungar, "Sound and Vibration Damping with Polymers," No. 424 ACS Symposium Series, Chapt. 17, (1989).

In another aspect of the present invention, a cured fluorene-containing epoxy resin is used in a three layer laminate comprising a base layer (vibratory solid object) to be damped, a viscoelastic layer, and a constraining layer. This application is referred to as a "constrained" viscoelastic layer, sometimes referred to as "shear" damping (Kerwin and Ungar, *supra.*).

Cured fluorene-containing epoxy resins are also useful in variations of the constrained viscoelastic layer configuration, such as a segmented constraining layer, multiple constrained layer treatments, and multiple, overlapping segmented layers.

Fluorene epoxies provide a class of materials that exhibit high performance damping capabilities for use at operating temperatures between about 120 to 275°C (at 1 Hz) and provide toughness over a much broader temperature range. In addition, these resins exhibit ductility and are resistant to moisture.

EP-A-439197 shows that heterogeneous compositions comprising low to medium crosslinked aromatic epoxides can be used as the viscoelastic component in many vibration damping constructions. The present invention as defined in claims 1 and 6 shows that such compositions, if they exhibit a microheterogeneous morphology and include in particular cured fluorene-containing epoxy resins, can be used as the viscoelastic component in both free and constrained-layer high temperature (> 100°C) damping constructions. Damping performance for these compositions can be further enhanced by controlling the degree of crosslinking and fluorene content. Optionally, other plastic and elastic components may be added to control the degree of crosslinking and fluorene content. Furthermore, due to the intrinsic low water uptake of the fluorene containing epoxide materials, they can advantageously be used in hot, humid environments.

Properties of vibration-damping materials are described in the literature. Nielsen, L.E., "Mechanical Properties of Polymers," pages 162-65, Reinhold Publishing Corp., New York, (1965) discloses that materials possessing maximum vibration-damping capability have storage moduli, G' , greater than 1MPa (10^7 dynes/cm²) but less than 1GPa (10^{10} dynes/cm²) at the use temperature. Furthermore, Rosen, S.L., "Fundamental Principles of Polymeric Materials for Practicing Engineers," pages 222-27, Barnes & Noble Inc., New York, (1971), shows that it is desirable for a vibration-damping material to have both a storage modulus and a loss tangent with values as high as possible.

Corsaro, R.D. and Sperling, L.H. (Eds.), "Sound and Vibration Damping with Polymers," ACS Symposium Series No. 424, American Chemical Society, Washington D.C. (1989) disclose general definitions, and concepts, as well as specific applications for viscoelastic vibration-damping materials.

As used in this application:

"complex modulus", designated by G^* , is expressed as $G^* = G' + iG''$;

"constrained layer" means a damping configuration comprising the component or structure to be damped, a viscoelastic layer, and a constraining layer;

"cure" and "polymerize" are used interchangeably in this application to indicate a chemical reaction, usually carried out with a catalyst, heat or light, in which a large number of relatively simple molecules combine to form a chain-like macromolecule;

"epoxy resin composition" is used to indicate uncured compositions, comprising polyepoxide, fluorene-containing epoxy, curing agents, fluorene containing curing agents and other components such as thermoplastics, thermosets and elastomers that can be cured to a "cured fluorene-containing epoxy resin";

"free layer" means a damping configuration comprising a viscoelastic layer bonded to the component or structure to be damped;

"loss modulus", designated by G'' , is a measure of the equivalent energy lost (as heat);

"storage modulus", designated by G' , is a measure of the equivalent energy stored elastically;

"thermoplastic" means a high polymer that softens when exposed to heat and returns to its original condition when cooled to room temperature; and

"thermoset" means a high polymer that solidifies or "sets" irreversibly when heated. This property is associated with a crosslinking reaction of the constituents.

Description of The Preferred Embodiment Of The Invention

5 Aromatic epoxides and in particular fluorene-containing networks exhibit good high temperature damping behavior from about 120°C to about 275°C measured at 1 Hz, and as such are suitable as the viscoelastic component in both free-layer and constrained-layer damping constructions. The temperature range and extent of damping of these compositions are dependent upon the particular aromatic epoxy selected and the fluorene content. The addition of ther-
10 mosets, thermoplastics and elastomers may also affect performance of the compositions.

The preferred polymer damping materials may comprise a combination of polymers and exhibit a microheterogeneous morphology where phase boundaries are diffuse and the minor phase (present in a lesser amount) has a size on the order of hundreds of angstroms. Damping materials that consist of two or more polymers and have macroscopically phase separated morphologies (on the order of micrometers) display damping performance at temperatures associated with the T_g 's of the individual polymers. Conversely, two or more polymers that are mutually soluble and form a single phase exhibit damping over a narrow temperature range, the same as a single polymer. Thus, in order to achieve damping over a broad temperature range a multicomponent system is required.

Traditionally, in macroscopically phase separated systems, component polymers have been chosen based on the temperature interval between their respective T_g 's so that their individual damping characteristics overlap. However, with a microheterogeneous-morphology, damping behavior covering a broad temperature range is possible since there is a gradation of T_g character resulting from intimate mixing at diffuse interface boundaries.

In general, damping materials are applied to structures and component parts in devices to attenuate resonant vibrations thereby reducing noise and vibrational fatigue. This is often accomplished by attaching a viscoelastic material of appropriate damping characteristics to the vibrating structure. Vibrational forces cause the viscoelastic material to undergo shear deformation where some of its inelastic deformation energy is converted to heat and then dissipated (mechanical hysteresis). Under cyclic loading, heat generated results in a temperature rise until heat generated per cycle is equal to heat dissipated through conduction, convection and radiation. The ability of a material to damp is measured by its ability to convert vibrational energy to heat energy.

In viscoelastic materials, the maximum amount of energy is dissipated as heat at the glass-rubber transition temperature (T_g). Effectiveness of a viscoelastic material in energy dissipation is evaluated by measuring its viscoelastic response to a periodic stress or strain. Results of dynamic mechanical tests are generally given in terms of elastic or storage modulus, G' , and viscous or loss modulus, G'' . G'' is the damping or energy dissipation term. The angle which reflects the time lag between applied strain and stress is known as delta (δ), and is defined by the ratio called the dissipation factor or loss factor.

35 $\tan(\delta)$ is a damping term and is a measure of the ratio of energy dissipated as heat to maximum energy stored in the material during one cycle of oscillation and can be defined as:

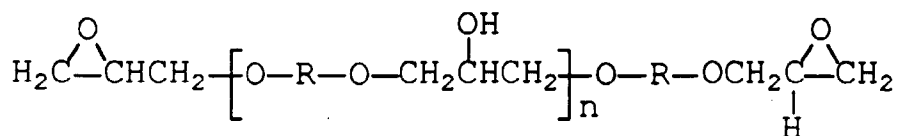
$$\tan(\delta) = \frac{G''}{G'}$$

40 Dynamic mechanical criteria used to evaluate high temperature damping performance are as follows:

1. $\tan(\delta)$ is above 100°C,
2. $\tan(\delta)$ is equal to or greater than 0.6, and
3. $1\text{MPa} < G' < 1\text{GPa}$

Surprisingly, it has been found incorporation of fluorene structures or fluorene segments into an epoxy produces a family of high performance epoxy resins that meet these requirements and are useful in high temperature damping applications. The fluorene moiety may be introduced into an epoxy via addition of a fluorene-containing epoxy or using a fluorene amine or a combination of both. Fluorene is a large rigid planar structure of tricyclic hydrocarbon, and it is believed this backbone structure contributes to producing tough, high T_g resins. Generally, thermosetting resins require high levels of crosslinking to achieve enhanced T_g characteristics. Consequently, those resins exhibit a reduction in toughness and damping performance. However, as demonstrated in the present invention, high T_g damping materials, containing the fluorene moiety are produced with low levels of crosslinking resulting in compositions having high temperature and high damping characteristics. Furthermore, the presence of the fluorene moiety in any amount results in high performance damping materials.

U.S. Patent No. 4,707,534, describes glycidyl ethers useful in the practice of the present invention and are represented generally by the formula



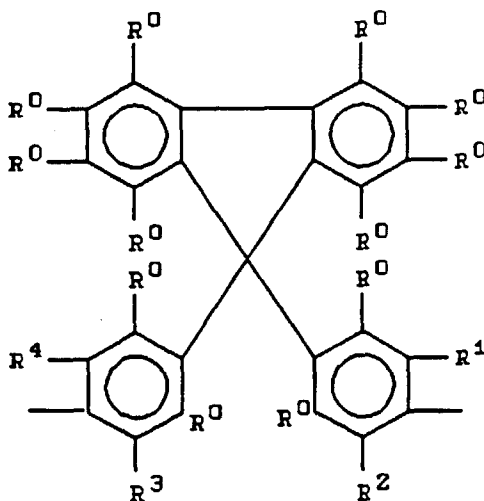
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wherein

n is zero or a number having a value of 1 to 3, preferably, n is zero, and

R is a divalent organic group having the formula in

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which

each R⁰

is independently selected from hydrogen and groups that are inert in the polymerization of epoxide group-containing compounds which are preferably selected from halogen, linear and branched alkyl groups having 1 to 6 carbon atoms, phenyl, nitro, acetyl, and trimethylsilyl; and

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each of R¹, R², R³, and R⁴

is independently selected from hydrogen, linear and branched alkyl groups having 1 to 6 carbon atoms, phenyl and halogen;

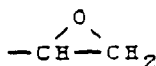
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with the provisos that at least one of R¹, R², R³, and R⁴ is a linear or branched alkyl group having 1 to 6 carbon atoms, a phenyl group, or halogen and the epoxy equivalent weight of the diglycidyl ether is at most about 500.

The curable composition of the invention contains at least 25 percent by weight (i.e., 25 to 100 weight percent), preferably 50 to 100 weight percent, of the above defined glycidyl ether and up to 75 percent, and more preferably 0 to 50 weight percent by weight, of other aromatic polyepoxide. Such aromatic polyepoxides are well known and are compounds in which there is present at least one aromatic ring structure, e.g. a benzene ring, and more than one epoxy group, e.g.

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In the composition, monoepoxide compounds may also be included. The aromatic polyepoxides preferably are the polyglycidyl ethers of polyhydric phenols, glycidyl esters of aromatic carboxylic acids, N-glycidylaminobenzenes, and glycidylamino-glycidyl-oxy-benzenes. More preferably the aromatic polyepoxides are the polyglycidyl ethers of polyhydric phenols. U.S. Patent No. 4,707,534 describes additional illustrative examples of diglycidyl epoxy resins.

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These fluorene epoxy (FEP) resins are also known to exhibit high temperature mechanical performance, high ductility and low equilibrium moisture uptake. See for example, Schultz et al., ACS Polymer Preprints, Vol. 29(1), June

1988 and Schultz et al., SAMPE 20th International Technical Conference, Minneapolis, MN, Sept 1988. However, they have not been used in either "free" viscoelastic layer or "constrained" viscoelastic layer applications. It has only been recently and surprisingly appreciated that their mechanical performance, and high ductility are applicable to use as vibration damping materials.

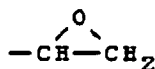
5 Dynamic mechanical experiments were performed on several aromatic epoxy and FEP compositions. G' was generally observed to be less than 10¹⁰ dynes/cm² in the glassy state and greater than about 10⁷ dynes/cm² in the rubbery region. The temperature interval over which effective damping was observed (Tan (δ) ≥ 0.6) for a given composition was approximately 28°C. Specific temperatures where maximum damping was observed were determined by the T_g of the resin composition.

10 Epoxy resins that display good damping characteristics can be prepared, in part, from fluorene epoxy. Alternatively, fluorene can be introduced into the epoxy backbone through an amine curative. The following composition demonstrates a conventional epoxy cured with a fluorene amine curative.

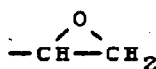
U.S. Patent No. 4,684,678 describes an epoxy resin composition useful in the practice of the present invention and comprises

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- (a) at least one aromatic polyepoxide, and
 - (b) at least one 9,9-bis(aminophenyl)fluorene (also referred to as "fluorene" herein) curing agent sufficient to provide in the range of 0.1 to 1.1 amino groups per epoxy group present in the aromatic polyepoxide.

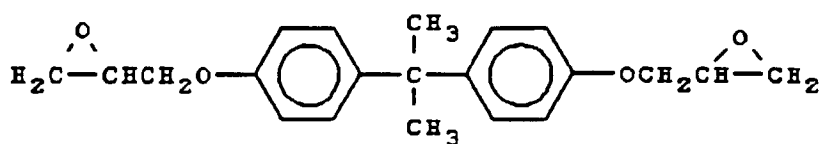
20 Within this application, the term "polyepoxide" means a molecule that contains more than one



group and the term "aromatic polyepoxide" means a molecule that contains more than one



35 group that are attached directly or indirectly to an aromatic nucleus such as a benzene, diphenyl, diphenyl-methane, diphenylpropane, or naphthalene nucleus, etc. During the curing of the "epoxy resin composition", the "polyepoxide", as exemplified by



45 and the curing agent, as exemplified by

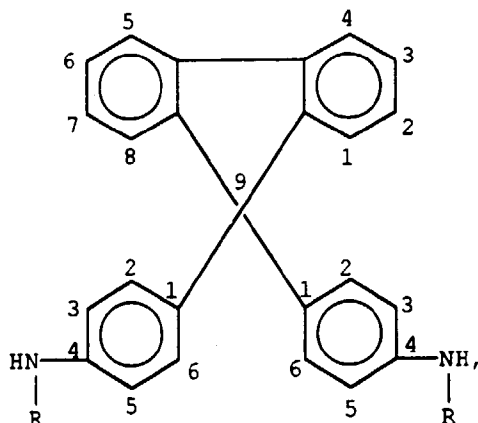
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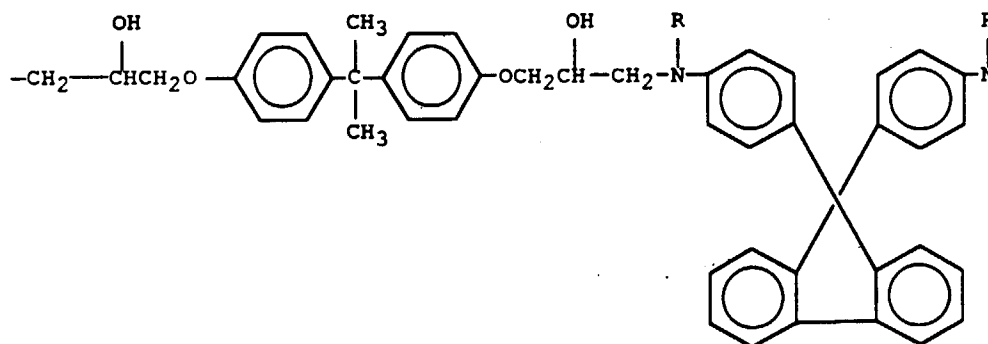


wherein R is as defined below, react to form a cured epoxy resin having units of

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The process aspect of the invention comprises the steps of (1) mixing the aromatic polyepoxides and the curing agent or agents and catalysts as described below to form a substantially uniform mixture and (2) heating the mixture for a time and at a temperature sufficient to cure the composition. While the curing reaction may take place slowly at room temperature, it preferably is brought about by heating the mixture to a temperature between 50°C and 300°C for a period of time from about one to about 18 hours or more. Furthermore, it is desirable to cure the mixture by heating in cycles such as, for example, 50° to 150°C for a time period of between 0.25 to 1.0 hour, 150° to 200°C for a time period of between 0.5 to 2.0 hours, and 175° to 250°C for a time period of between 1.0 to 5.0 hours.

U.S. Patent No. 4,684,678 describes additional illustrative examples of epoxy resin compositions.

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Epoxy resin curing typically involves dispersing amine curatives in the epoxy resin. Curatives need only be added in an amount sufficient to affect curing of the epoxy resin composition. For example, the curative may be added in stoichiometric excess or may be used in less than stoichiometric amounts in combination with a catalyst, such as, Lewis acids, tertiary amines and imidazoles. Amine curatives suitable for use in the present invention include:

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- 9,9-bis(4-aminophenyl)fluorene,
- 4-methyl-9,9-bis-(4-aminophenyl)fluorene,
- 4-chloro-9,9-bis-(4-aminophenyl)fluorene,
- 2-ethyl-9,9-bis-(4-aminophenyl)fluorene,
- 2-iodo-9,9-bis-(4-aminophenyl)fluorene,
- 3-bromo-9,9-bis-(4-aminophenyl)fluorene,
- 9-(4-methylaminophenyl)-9-(4-ethylaminophenyl)fluorene,
- 1-chloro-9,9-bis-(4-aminophenyl)fluorene,
- 2-methyl-9,9-bis-(4-aminophenyl)fluorene,
- 2,6-dimethyl-9,9-bis-(4-aminophenyl)fluorene,
- 1,5-dimethyl-9,9-bis-(4-aminophenyl)fluorene,
- 2-fluoro-9,9-bis-(4-aminophenyl)fluorene,
- 1,2,3,4,5,6,7,8-octofluoro-9,9-bis-(4-aminophenyl)fluorene,
- 2,7-dinitro-9,9-bis-(4-aminophenyl)fluorene,

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2-chloro-4-methyl-9,9-bis-(4-aminophenyl)fluorene,
 2,7-dichloro-9,9-bis-(4-aminophenyl)fluorene,
 2-acetyl-9,9-bis-(4-aminophenyl)fluorene,
 2-methyl-9,9-bis-(4-methylaminophenyl)fluorene,
 2-chloro-9,9-bis-(4-ethylaminophenyl)fluorene,
 2-t-butyl-9,9-bis-(4-methylaminophenyl)fluorene,

The use of combinations of fluorene-containing diprimary and dissecondary amines is preferred and allows the preparation of fluorene-containing networks that are particularly useful as damping materials. Dissecondary amines act as chain extenders and tend to produce a linear polymer while diprimary amines contribute to crosslinking. Fluorene content in the epoxy resin composition can be controlled by curing with varying ratios of diglycidyl ether of bisphenol-A (DGEBA) and a fluorene containing resin as well as by using fluorene amine curatives. T_g can be controlled via cure chemistry, such as adjusting the extent of crosslinking with the diprimary amine and/or by changing the fluorene concentration.

Fluorene-containing epoxy compositions may also be modified by the addition of thermoplastics, thermosets, and elastomers. Thermoplastics that are suitable may include for example, polyphenylene sulfone, polybenzimidazole, polyether sulfone, polyester, polyimide, polyetherimide, polyphenylene oxide, polysulfone, acrylate, and methacrylate. For example, polyetherimide (ULTEM™, General Electric) enhances damping properties of the resin. It can also be used to control viscosity. FEP compositions can be rubber toughened by dispersing conventional rubber-toughening agents, such as core/shell rubber in the epoxy prior to adding the curatives, see U.S. Patent No. 4,684,678. This potentially improves the composite's low-end temperature damping characteristics. It is this flexibility in cure chemistry that allows formulation of compositions to address specific high temperature damping needs.

Other useful materials that may be blended into the composition include, but are not limited to, fillers, pigments, fibers, woven and nonwoven fabrics, foaming agents, antioxidants, stabilizers, fire retardants, and viscosity adjusting agents.

A free layer damping construction may be prepared according to processes known to those in the art and may comprise the following steps:

1. coating a release liner with a layer of epoxy resin composition,
2. curing the composition into a viscoelastic layer,
3. fixedly mounting the viscoelastic layer to a vibratory article that is to be damped, and
4. removing the release liner.

Alternatively, a free layer damping construction may be prepared as follows:

1. coating a vibrating article that is to be damped with a layer of an epoxy resin composition, and
2. curing the composition into a viscoelastic layer in situ to form a free layer vibration-damping construction.

Since the viscoelastic layer generally has some adhesive properties, the cured epoxy resin could be readily adhered to the vibratory article without the use of an adhesive. However, it is sometimes desirable to use a thin layer (for example, 20-50 μm) of a high-modulus adhesive, such as an acrylic adhesive or an epoxy adhesive, to bond the viscoelastic layer to the vibratory article.

The layer thickness of a free-layer damping construction is generally greater than for a constrained layer construction, since damping performance of the free-layer construction is a function of the layer thickness, see Kerwin and Ungar, *supra*.

Vibration damping laminates containing fluorene-containing networks may be prepared according processes well known in art, for example, according to the steps of either Method I or Method II as described in U.S. Patent No. 4,447,493.

For example, Method I provides the following steps:

1. coating a release liner with a layer of an epoxy resin composition;
2. curing the composition into a viscoelastic layer,
3. transferring the viscoelastic layer from the release liner to a substrate,
4. adhering the viscoelastic layer into the substrate to form the constrained-layer vibration-damping construction, and
5. fixedly mounting the constrained layer vibration-damping construction to a vibratory article to be damped.

Alternatively, Method II provides the following steps:

1. coating a substrate with a layer of an epoxy resin composition;
2. curing the composition to a viscoelastic layer in situ onto a substrate to form the constrained-layer vibration-damping construction; and
3. fixedly mounting the constrained-layer vibration-damping construction to a vibratory article to be damped.

5 The constrained-layer construction can be mechanically or adhesively affixed to the vibratory article that is to be damped. Since the viscoelastic layer generally has some adhesive properties, the cured resin can usually be adhered to a stiff layer or substrate without the use of an adhesive. It is sometimes desirable, however, to use a thin layer (for example, 20-50 μ m) of high-modulus adhesive, such as an acrylic adhesive or an epoxy adhesive, to bond the viscoelastic layer to a solid article which can be, for example, an oil pan, a valve cover, or a transmission housing.

10 For most applications, the viscoelastic layer is a coating having a thickness of at least 0.01 mm up to about 100 mm, preferably 0.025 to 100 mm, and most preferably 0.05 to 100 mm. The coating can be applied by any of the techniques known in the art such as by spray, dip, knife, or curtain coating.

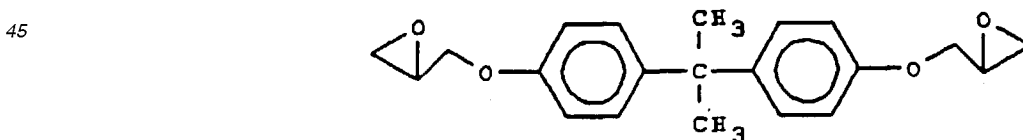
15 A stiff layer or a substrate is an essential part of constrained-layer vibration-damping constructions. A suitable material for a substrate has a stiffness of 0.40 (relative to stainless steel) as defined in "Handbook of Tables for Applied Engineering Science", ed. Bolz, R. E. et al., CRC Press, Cleveland, Ohio, page 130 (1974). The desired stiffness of the substrate is varied by adjusting the thickness of the layer, for example from about 25 micrometers to 5 centimeters, depending on the modulus of the substrate. Examples of suitable materials include metals such as iron, steel, nickel, aluminum, chromium, cobalt and copper, and alloys thereof; stiff thermoplastic materials, such as polystyrene, polyvinyl chloride, polyurethane, polyphenyl sulfide, and polycarbonate; stiff thermoset materials; fiber-reinforced thermoplastics; fiber-reinforced thermosets, such as epoxies, phenolics; ceramic fiber; and metal fiber-reinforced polyester, glasses, and ceramics.

20 Objects and advantages of this invention are further illustrated by the following examples, but the particular materials and amounts thereof recited in these examples, as well as other conditions and details, should not be construed to unduly limit this invention. All materials are commercially available except where stated or otherwise made apparent. Measurements reported hereinbelow were made using a Rheometrics 700 RDA operating in dynamic temperature step mode with a torsion rectangular specimen geometry. Results were analyzed in terms of G' , G'' and $\tan(\delta)$ as a function of temperature.

30 Examples

Dynamic mechanical analysis (DMA) results are outlined below and illustrate a number of fluorene-containing networks that exhibited good high temperature damping characteristics. As a result, they are suitable as the viscoelastic component in both free-layer and constrained-layer damping constructions. For high temperature applications using aromatic epoxies and FEPS, the temperature range over which $\tan(\delta) > 0.6$ spans from about 120° to about 275°C. The temperature interval over which effective damping was observed ($\tan(\delta) = 0.6$) for a given composition was approximately 28°C. Specific temperatures where maximum damping was observed were determined by the, G^* of the particular composition. G' is generally observed to be less than 1 GPa (10^{10} dynes/cm²) in the glassy state and greater than about 1 MPa (10^7 dynes/cm²) in the rubbery region. The Tables listed below show the effect of compositional and cure chemistry changes on the amplitude of $\tan(\delta)$, on the temperature where $\tan(\delta)$ is a maximum and on the temperature interval where $\tan(\delta)$ is equal to 0.6.

Nomenclature and chemical structures used to identify the compositions are as follows:

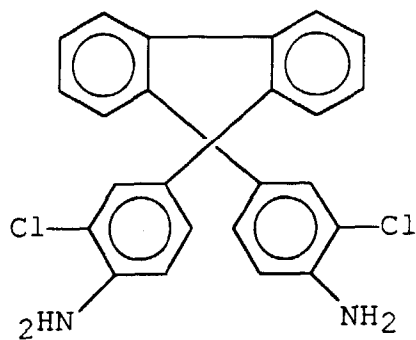


50 **DER 332**

DGEBA (diglycidylether of bisphenol-A)
(DER 332™, available from Dow Chemical)

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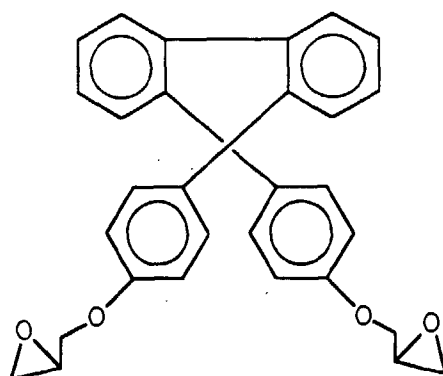


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CAF
(diprimary amine curative)

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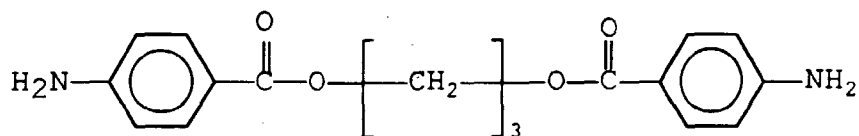


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FEP (fluorene epoxy)
(HPT 1079, available from Shell Chemical)

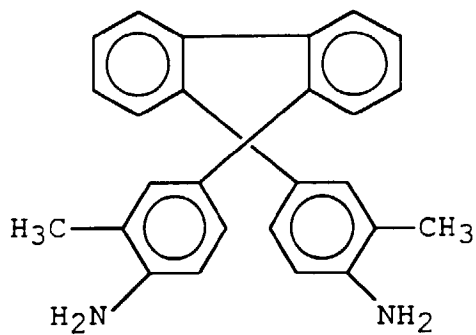
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PDAB
(diprimary amine curative) (available from Air Products)

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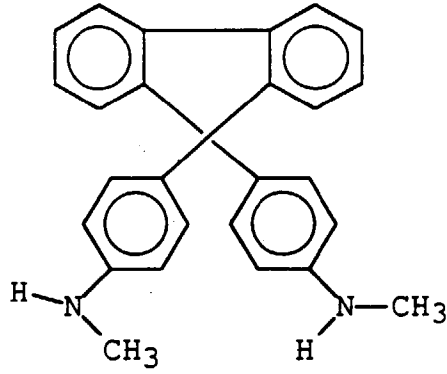


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OTBAF
(diprimary amine curative)

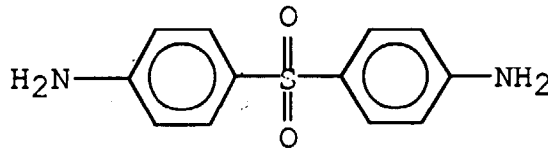
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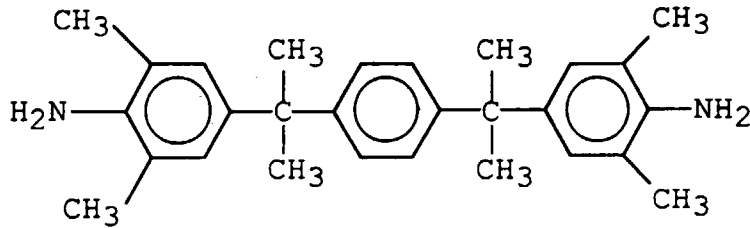
15 BMAF
(dissecondary amine curative)

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25 DDS
(diprimary amine curative)

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HPT-1062™ (diprimary amine curative)
(available from Shell Chemical)

40 Other compositions useful in the present invention include the following:

MFEP methyl fluorene epoxy (U.S. Patent 4,707,534)

45 MY0510 trifunctional epoxy (available from Ciba Geigy)

CIS RUB EXL 2691™ core/shell rubber (available from Rohm & Haas)

EPX amine terminated polytetramethylene oxide rubber having a number molecular weight of 100,000

50 IBA polyisobornyl acrylate

IBMA polyisobornyl methacrylate

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The equivalent weight of the preferred compounds are as follows:

	Epoxy equivalent weight
DGEBA	175 grams/equivalents

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(continued)

	Epoxy equivalent weight
FEP	250 (average)
MFEB 252	(theoretical)
MY0510	100

	NH equivalent weights
DDS	62
PDAB	78.5
CAF	108
HPT 1062™	100
OTBAF	94

In the following examples, the epoxy resin has an equivalent of 1. The weight in grams for the constituents of the composition for preparing the following examples can be calculated as follows:

$$\frac{\text{grams of epoxy composition}}{\text{equivalents}} = \text{grams/equivalents of epoxy composition} \quad (1)$$

$$\text{equivalents} \times \% \text{ eq. of curative needed} = \text{equivalents of curative} \quad (2)$$

$$\text{equivalents of curative} \times \text{grams/equivalents of curative} = \text{grams of curative} \quad (3)$$

Generically, the compositions tested below were prepared by dispersing crystalline amine curatives in the epoxy resin composition with a high speed mixer. The dispersion was then heated to 177°C for approximately four hours. See U.S. Patent 4,684,678, for specific details.

EXAMPLES 1 - 8

Effect of crosslink density on high temperature damping characteristics of aromatic epoxides was assessed using a series of DGEBA epoxies (DER 322™) cured with three different diaromatic amines (DDS, PDAB and CAF) at varying concentrations. Note, stoichiometric levels of diprimary amine curative to epoxy produce tightly crosslinked fluorene-containing networks while higher ratios of DGEBA to diaromatic amines produced more lightly crosslinked thermosets.

Table 1

Composition Epoxy/Curative	Cross- Linked	% eq wt. Curative	Tan(δ) _{MAX}	Temp ($^{\circ}$ C) at Tan(δ) _{MAX}	Temp Range ($^{\circ}$ C) at Tan(δ) = 0.6
DER332(1.0)/DDS	High	1.00	0.75	204	10.0
DER332(1.0)/DDS	Medium	1.35	1.32	194	17.0
DER332(1.0)/DDS	Light	1.65	1.56	179	16.3
DER332(1.0)/PDAB	High	1.00	0.77	170	10.0
DER332(1.0)/PDAB	Light	1.65	1.57	120	17.0
DER332(1.0)/CAF	High	1.00	0.96	194	16.0
DER332(1.0)/CAF	Medium	1.35	1.64	189	20.0
DER332(1.0)/CAF	Light	1.65	1.91	174	23.0

Table 1 shows that damping was enhanced as the level of crosslinking was reduced. That is, the amplitude of Tan(δ) [Tan(δ)_{max}] increased, the temperature at which Tan(δ) is a maximum decreased and the temperature interval where Tan(δ) = 0.6 increased as the molecular weight between crosslinks was reduced.

EXAMPLES 9 - 12

Gain in damping properties as a consequence of reducing crosslink density is further illustrated for DGEBA/FEP compositions as shown in Table 2. Again, the more lightly crosslinked thermosets exhibited the greatest damping over the widest temperature range.

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Table 2

Composition (65/25/10)	Cross- Linked	% eq wt. CAF	Tan(δ) _{MAX}	Temp (°C) at Tan(δ) _{MAX}	Temp Range (°C) at Tan(δ) = 0.6
DER332/FEP/MY0510	High	1.00	0.75	204	10.0
DER332/FEP/MY0510	Medium	1.35	1.43	208.8	13.3
DER332/FEP/MY0510	Light	1.50	1.60	210	21.0
DER332/FEP/MY0510	Light	1.65	1.68	201.1	23.8

EXAMPLES 13 - 20

For a given crosslink density, damping is further enhanced when fluorene is contained in the epoxide composition

as illustrated in Table 3 (Part A). Fluorene can be incorporated in the epoxide composition via a fluorene amine curative. Alternatively, fluorene was incorporated by replacing all or part of DGEBA with a fluorene containing epoxide resin. Table 3 (Part A) shows that the damping factor, $\text{Tan}(\delta)$, was greatest in amplitude and covered the broadest temperature range for compositions containing more than 73% fluorene. Further 100% fluorene containing thermoset resin compositions are shown in Table 3 (Part B). These resins exhibited the highest temperature damping properties observed.

Table 3
PART A

Composition Epoxy/Curative	% eq. wt. curativ e	% by wt. Fluorene	$\text{Tan}(\delta)$ MAX	Temp (°C) at $\text{Tan}(\delta)$ MAX	Temp Range (°C) at $\text{Tan}(\delta) = 0.6$
DER332/DDS	1.65	0	1.56	179	16.3
DER332/PDAB	1.65	0	1.57	120	17.0
DER332/HPT 1062™	1.65	0	1.65	140	16.0
DER332/CAF	1.65	50	1.91	174	23.0
[DER332/FEP(50:50)] /CAF	1.65	73	2.10	194	24.0
[DER332/FEP(50:50)] /CAF	1.65	86	2.20	214	28.0
PART B					
FEP/CAF	1.65	100	2.20	219	25.0
MFEP/CAF	1.35	100	2.38	245.4	21.5

EXAMPLES 21 - 22

5 Since epoxide damping characteristics are impacted by crosslink content, curing the epoxides with both a diprimary amine (CAF) and a dissecondary amine (BMAF) provide another means for controlling damping characteristics, as shown in Table 4. Since BMAF behaves as a chain extender, compositions having higher BMAF content had a lesser degree of crosslinking. As shown previously, the lower the crosslink content, the lower the temperature of the Tan(δ) peak and the greater its magnitude.

10 EXAMPLES 23 - 27

Damping behavior of the epoxide resins can be further enhanced with the addition of high T_g thermoplastics. In Table 5, the effect of adding polyetherimide (ULTEM™, available from General Electric) on the damping characteristics of the epoxy composition is shown. The major effect of adding 6% polyetherimide to the epoxy resin was a doubling of the temperature interval where effective damping was observed. Additionally, some increase in magnitude of Tan (δ) was observed with addition of polyetherimide. An unusually large amplitude was observed for the composition containing 12% polyetherimide.

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Table 4

Composition (65/25/10)	CAF/BMAF 1.05% eq wt.	Tan(δ) _{MAX}	Temp (°C) at Tan(δ) _{MAX}	Temp Range (°C) at Tan(δ) = 0.6
DER332/FEP/MY0510	0.45/0.60	2.00	193.1	22.7
DER332/FEP/MY0510	0.72/0.35	1.67	198.9	22.1

Table 5

Composition (65/35/10)	% eq wt. CAF	% by wt. ULTEM ^m	Tan(δ) _{MAX}	Temp (°C) at Tan(δ) _{MAX}	Temp Range (°C) at Tan(δ) = 0.6
DER332/FEP/MY0510	1.35	0	1.43	208.8	13.3
DER332/FEP/MY0510	1.35	6	1.72	209.4	26.1
DER332/FEP/MY0510	1.35	9	1.63	207.7	25.5
DER332/FEP/MY0510	1.35	12	2.42	207.7	26.1
DER332/FEP/MY0510	1.35	15	1.74	207.7	26.2

EXAMPLES 28 - 29

Similar to what was observed in Examples 23-27, the damping performance of aromatic epoxides was enhanced with the addition of high temperature acrylates, such as IBA and IBMA as shown in Table 6.

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Table 6

Composition Epoxy/ Acrylate (88/12)	% eq wt. CAF	Tan(δ) MAX	Temp ($^{\circ}$ C) at Tan(δ) MAX	Temp Range ($^{\circ}$ C) at Tan (δ) = 0.6
DER332/IBA	1.5	1.72	190	23.0
DER332/IBMA	1.5	1.47	182.2	26.3

EXAMPLES 30 - 37

Soluble elastomers (EPX) that phase separated during the curing process and core/shell rubber particles were added to epoxy resin compositions to improve their adhesive characteristics and toughness. This also improved the low-end temperature characteristics of the cured resins. Incorporation of these elastomers tends to reduce high temperature damping performance of resin when the weight percent of the elastomers is too high. However, as shown in Tables 7 and 8, good damping performances were observed for cured resins having an elastomer content up to 15% by weight. As discussed above, crosslink density was controlled by adjusting the ratio of diprimary to dissecondary amine curative.

Table 7

Epoxy Composition (50/50)	OTBAF/BMAF 1.05% eq wt.	% by wt. EPX	Tan(δ) _{MAX}	Temp (°C) at Tan(δ) _{MAX}	Temp Range (°C) at Tan(δ) = 0.6
DER332/FEP	0.55/0.50	5	1.65	210.0	22.6
DER332/FEP	0.55/0.50	10	1.55	208.5	21.3
DER332/FEP	0.55/0.50	15	1.31	203.9	22.1
DER332/FEP	0.45/0.60	10	2.63	203.3	25.1

Table 8

Epoxy Composition	OTBAF/BMAF 1.05% eq wt.	% by wt. EPX	Tan(δ) _{MAX}	Temp (°C) at Tan(δ) _{MAX}	Temp Range (°C) at Tan(δ) = 0.6
DER332	1.05/0.0	0	1.28	232.8	18.4
DER332	1.05/0.0	10	1.13	229.4	19.2
DER332	0.55/0.50	0	1.99	192.7	21.7
DER332	0.45/0.60	10	2.03	186.1	21.1

EXAMPLES 38 - 39

Two fluorene-containing epoxy compositions were prepared by mixing the following ingredients:

	A	B
PY 306	180 grams	180 grams
OTBAF	154 grams	154 grams
EPX†	37 grams	---
ULTEM [™] ‡	---	37 grams

† polytetramethylene oxide diprimary amine (MW = 10,000), available from 3M, Co.
 ‡ polyetherimide, available from General Electric

The components of formulation A and B were vigorously mixed until a smooth homogeneous mixture was obtained. The mixtures were then hot melt coated onto films 0.127-0.153 mm (5 to 6 mils) thick. A constrained layer construction was then prepared by placing a layer of hot melt coated film on the surface of a 153 mm by 330 mm (6 inch by 13 inch) steel sheet. The steel sheets were sandwiched together after placing a thin nylon scrim on one surface. The scrim was used only to prevent the resin from flowing out of the sandwich during the curing process. The sandwich construction was placed in an oven and cured at 177°C for approximately two hours. The resulting constrained layer construction had 0.203 mm (8 mils) of resin bonded to the steel sheets. The shear strength of the construction was 6.89 MPa (1000 pounds/square inch).

The process was repeated as recited substituting formulation B for formulation A.

Various modifications and alterations of this invention will become apparent to those skilled in the art, and it should be understood that this invention is not to be unduly limited to the illustrative embodiments set forth hereinabove.

Claims

1. A method for damping the vibration of a vibrating solid article at temperatures in the range of 120° to 275°C at 1 Hz, wherein said method comprises providing a viscoelastic layer construction with at least one layer of a damping material, characterized by exhibiting a microheterogenous morphology in which the minor phase has a size on the order of tens of nanometres (hundreds of angstroms) and comprising a cured fluorene-containing epoxy resin.
2. The method according to claim 1, wherein said cured fluorene-containing epoxy resin has a glass transition temperature in the range of 120°C to 275°C at 1 Hz.
3. The method according to claim 1, wherein said viscoelastic layer construction is a free-layer construction.
4. The method according to claim 1, wherein said viscoelastic layer construction is a constrained-layer construction comprising at least one substrate having a stiffness relative to stainless steel of at least 0.4 coated with at least one continuous layer of a cured fluorene-containing epoxy resin.
5. The method according to claim 4, wherein said substrate is selected from the group consisting of metals, stiff thermoplastic materials, stiff thermoset materials, fiber-reinforced thermoplastics, fiber-reinforced thermosets, ceramic fibers, glass, ceramics, and metal fiber-reinforced polyester.
6. A damping construction for the damping of mechanical vibrations comprising at least one layer of viscoelastic material exhibiting a microheterogenous morphology in which the minor phase has a size on the order of tens of nanometres (hundreds of angstroms) which is applied to a vibratory solid article, said viscoelastic material comprising a cured fluorene-containing epoxy resin, optionally including a thermoplastic, thermoset or elastomer.
7. The damping construction according to claim 6, wherein a substrate is applied over said viscoelastic material wherein said substrate is selected from the group consisting of metals, stiff polymeric materials, fiber-reinforced thermoplastics, fiber-reinforced thermosets, ceramic fibers, glass, ceramics and metal fiber-reinforced polyester.
8. The damping construction according to claim 6, wherein said viscoelastic material is the reaction product of a curable fluorene-containing composition comprising:

- a. a glycidyl epoxide, wherein said glycidyl epoxide may be fluorene containing; and
- b. aromatic amine curative in an amount sufficient to cure the mixture, wherein said amine curative may be fluorene-containing,

5 with the proviso that at least one of said glycidyl epoxide or said aromatic amine curative is fluorene containing.

9. The damping construction according to claim 6,
wherein said thermoplastic is selected from the group consisting of polyphenylene sulfone, polybenzimidazole, polyether sulfone, polyester, polyimide, polyetherimide polyphenylene oxide, polysulfone, acrylate, and methacrylate.

10. The damping construction according to claim 6,
wherein said elastomer is a rubber toughening agent.

11. A method according to claim 3 comprising:

- a. coating a release liner with a layer of fluorene-containing epoxy resin composition;
- b. curing said composition into a viscoelastic layer;
- c. fixedly mounting said viscoelastic layer to a vibratory article that is to be damped; and
- d. removing said release liner.

12. A method according to claim 3 comprising:

- a. coating a vibrating article that is to be damped with a layer of a fluorene-containing epoxy resin composition, and
- b. curing said composition into a viscoelastic layer in situ to form a free layer vibration-damping construction.

13. A method according to claim 1 wherein said viscoelastic layer construction is a constrained layer construction comprising:

- a. coating a release liner with a layer of fluorene-containing epoxy resin composition;
- b. curing said composition into a viscoelastic layer;
- c. transferring said viscoelastic layer from said release liner to a substrate, so as to adhere said viscoelastic layer to said substrate to form said constrained-layer vibration-damping construction; and
- d. fixedly mounting the constrained layer vibration-damping construction to a vibratory article that is to be damped.

14. A method according to claim 1 wherein said viscoelastic layer construction is a constrained layer construction comprising:

- a. coating a substrate with a layer of an epoxy resin composition;
- b. curing the composition into a viscoelastic layer in situ onto a substrate to form the constrained-layer vibration-damping construction; and
- c. fixedly mounting the constrained-layer vibration-damping construction to a vibratory article to be damped.

Patentansprüche

1. Verfahren zur Dämpfung der Vibration eines vibrierenden festen Gegenstandes bei Temperaturen im Bereich von 120° bis 275°C bei 1 Hz, wobei das Verfahren die Bereitstellung einer viskoelastischen Schichtkonstruktion mit mindestens einer Schicht eines Dämpfungsmaterials umfaßt, dadurch gekennzeichnet, daß es eine mikroheterogene Morphologie aufweist, wobei die Nebenphase eine Größenordnung von einigen zehn Nanometern (einigen hundert Angström) hat, und daß es ein gehärtetes fluorenhaltiges Epoxidharz umfaßt.
2. Verfahren nach Anspruch 1, wobei das gehärtete fluorenhaltige Epoxidharz bei 1 Hz eine Glasübergangstemperatur im Bereich von 120°C bis 275°C hat.
3. Verfahren nach Anspruch 1, wobei die viskoelastische Schichtkonstruktion eine Konstruktion mit freier Schicht ist.

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4. Verfahren nach Anspruch 1, wobei die viskoelastische Schichtkonstruktion eine Konstruktion mit Abdeckschicht ist, umfassend mindestens ein Substrat mit einer Steifigkeit von mindestens 0.4, bezogen auf Edelstahl, das mit mindestens einer geschlossenen Schicht eines gehärteten fluorenhaltigen Epoxidharzes beschichtet ist.
- 5 5. Verfahren nach Anspruch 4, wobei das Substrat aus Metallen, starren thermoplastischen Materialien, starren duroplastischen Materialien, faserverstärkten Thermoplasten, faserverstärkten Duroplasten, keramischen Fasern, Glas, Keramik und mit Metallfasern verstärktem Polyester ausgewählt ist.
- 10 6. Dämpfende Konstruktion zur Dämpfung mechanischer Vibrationen, umfassend mindestens eine Schicht eines viskoelastischen Materials, das eine mikroheterogene Morphologie aufweist, wobei die Nebenphase eine Größenordnung von einigen zehn Nanometern (einigen hundert Angström) hat, die an einem vibrierenden festen Gegenstand angebracht wird, wobei das viskoelastische Material ein gehärtetes fluorenhaltiges Epoxidharz umfaßt, das gegebenenfalls ein Thermoplast, ein Duroplast oder ein Elastomer einschließt.
- 15 7. Dämpfende Konstruktion nach Anspruch 6, wobei über dem viskoelastischen Material ein Substrat angebracht wird, wobei das Substrat aus Metallen, starren polymeren Materialien, faserverstärkten Thermoplasten, faserverstärkten Duroplasten, keramischen Fasern, Glas, Keramik und mit Metallfasern verstärktem Polyester ausgewählt ist.
- 20 8. Dämpfende Konstruktion nach Anspruch 6, wobei das viskoelastische Material das Umsetzungsprodukt einer härtbaren fluorenhaltigen Zusammensetzung ist, umfassend:
- 25 a. ein Glycidylepoxid, wobei das Glycidylepoxid Fluoren enthalten kann, und
b. ein aromatisches Aminhärtungsmittel in einer Menge, die ausreicht, um das Gemisch zu härten, wobei das Aminhärtungsmittel Fluoren enthalten kann, mit der Maßgabe, daß von dem Glycidylepoxid oder dem aromatischen Aminhärtungsmittel mindestens eines Fluoren enthält.
- 30 9. Dämpfende Konstruktion nach Anspruch 6, wobei der Thermoplast aus Polyphenylensulfon, Polybenzimidazol, Polyethersulfon, Polyester, Polyimid, Polyetherimid, Polyphenylenoxid, Polysulfon, Acrylat und Methacrylat ausgewählt ist.
- 35 10. Dämpfende Konstruktion nach Anspruch 6, wobei das Elastomer ein Modifizierungsmittel für Kautschuk ist.
11. Verfahren nach Anspruch 3, umfassend:
- 40 a. Beschichten einer Trennschicht mit einer Schicht einer fluorenhaltigen Epoxidharzzusammensetzung;
b. Härten der Zusammensetzung zu einer viskoelastischen Schicht;
c. festes Anbringen der viskoelastischen Schicht an einem vibrierenden Gegenstand, der gedämpft werden soll und
d. Entfernen der Trennschicht.
12. Verfahren nach Anspruch 3, umfassend:
- 45 a. Beschichten eines vibrierenden Gegenstands, der gedämpft werden soll, mit einer Schicht einer fluorenhaltigen Epoxidharzzusammensetzung und
b. Härten der Zusammensetzung in situ zu einer viskoelastischen Schicht, wobei eine vibrationsdämpfende Konstruktion mit freier Schicht entsteht.
- 50 13. Verfahren nach Anspruch 1, wobei die viskoelastische Schichtkonstruktion eine Konstruktion mit Abdeckschicht ist, umfassend:
- 55 a. Beschichten einer Trennschicht mit einer Schicht einer fluorenhaltigen Epoxidharzzusammensetzung;
b. Härten der Zusammensetzung zu einer viskoelastischen Schicht;
c. Übertragen der viskoelastischen Schicht von der Trennschicht auf ein Substrat, wobei die viskoelastische Schicht am Substrat befestigt wird und die vibrationsdämpfende Konstruktion mit Abdeckschicht bildet und
d. festes Anbringen der vibrationsdämpfenden Konstruktion mit Abdeckschicht an einem vibrierenden Gegenstand, der gedämpft werden soll.

14. Verfahren nach Anspruch 1, wobei die viskoelastische Schichtkonstruktion eine Konstruktion mit Abdeckschicht ist, umfassend:
- a. Beschichten eines Substrats mit einer Schicht einer Epoxidharzzusammensetzung;
 - b. Härten der Zusammensetzung in situ auf einem Substrat zu einer viskoelastischen Schicht, wobei die vibrationsdämpfende Konstruktion mit Abdeckschicht entsteht, und
 - c. festes Anbringen der vibrationsdämpfenden Konstruktion mit Abdeckschicht an einem vibrierenden Gegenstand, der gedämpft werden soll.

Revendications

1. Un procédé pour amortir les vibrations d'un article solide vibrant à des températures dans la gamme de 120 à 275°C à 1 Hz, ce procédé consistant à prévoir dans une construction à couche viscoélastique au moins une couche d'un matériau d'amortissement, caractérisé en ce qu'il présente une morphologie microhétérogène dans laquelle la phase mineure présente une dimension de l'ordre de quelques dixièmes de nanomètres (centaines d'Angstroms) et comportant une résine époxy, réticulée, renfermant du fluorène.
2. Le procédé selon la revendication 1, dans lequel ladite résine époxy réticulée, renfermant du fluorène, présente une température de transition vitreuse dans la gamme de 120 à 275°C à 1 Hz.
3. Le procédé selon la revendication 1, dans lequel la construction à couche viscoélastique est une construction à couche libre.
4. Le procédé selon la revendication 1, dans lequel ladite construction à couche viscoélastique est une construction à couche contrainte, qui comporte au moins un substrat présentant une rigidité relative par rapport à l'acier inoxydable d'au moins 0,4, revêtu d'au moins une couche continue d'une résine époxy réticulée, qui renferme du fluorène.
5. Le procédé selon la revendication 4, dans lequel le substrat est choisi dans le groupe constitué des métaux, des matériaux thermoplastiques rigides, des matériaux thermo-durcis rigides, des matériaux thermoplastiques renforcés par des fibres, des matériaux thermodurcis renforcés par des fibres, des fibres céramiques, du verre, des produits céramiques et des polyesters renforcés par des fibres métalliques.
6. Une construction d'amortissement pour l'amortissement des vibrations mécaniques comportant au moins une couche de matériau viscoélastique, qui présente une morphologie microhétérogène dans laquelle la phase mineure présente une dimension de l'ordre de quelques dixièmes de nanomètres (centaines d'Angstroms), qui est appliquée sur un article solide vibratoire, ce matériau viscoélastique comportant une résine réticulée, renfermant du fluorène, qui comporte facultativement une matière thermoplastique, une matière thermodurcie ou un élastomère.
7. La construction d'amortissement selon la revendication 6, dans laquelle un substrat est appliqué sur ledit matériau viscoélastique, ce substrat étant choisi parmi le groupe constitué des métaux, des matériaux polymères rigides, des matières thermoplastiques renforcés par des fibres, des matières thermodurcies renforcées par des fibres, des fibres céramiques, du verre, des produits céramiques et des polyesters renforcés par des fibres métalliques.
8. La construction d'amortissement selon la revendication 6, dans laquelle ledit matériau viscoélastique est le produit de réaction d'une composition réticulable, renfermant du fluorène, qui comporte:
 - a. un époxyde glycidyle, cet époxyde glycidyle pouvant renfermer du fluorène; et
 - b. une amine aromatique, apte à réticuler, en une quantité suffisante pour réticuler le mélange, ladite amine susceptible de réticuler pouvant renfermer du fluorène, sous la condition qu'au moins un parmi ledit époxyde glycidyle et ladite amine aromatique apte à réticuler renferme du fluorène.
9. La construction d'amortissement selon la revendication 6, dans laquelle ladite matière thermoplastique est choisie dans le groupe constitué d'une polyphénylène sulfone, d'un polybenzimidazole, d'une polyéther sulfone, d'un polyester, d'un polyimide, d'un oxyde polyphénylène/polyétherimide, d'une polysulfone, d'un acrylate et d'un méthacrylate.

10. La construction d'amortissement selon la revendication 6, dans laquelle ledit élastomère est un agent de vulcanisation du caoutchouc.

11. Un procédé selon la revendication 3, comportant:

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- a. le revêtement d'une couche amovible par une couche d'une composition de résine époxy, qui renferme du fluorène;
 - b. la réticulation de ladite composition en une couche viscoélastique;
 - c. le montage solide de ladite couche viscoélastique sur un article vibratoire qui doit être amorti; et
 - d. l'élimination du revêtement amovible.
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12. Un procédé selon la revendication 3, comportant:

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- a. le revêtement d'un article vibrant qui doit être amorti au moyen d'une couche d'une composition de résine époxy renfermant du fluorène, et
 - b. la réticulation de ladite composition en une couche viscoélastique in situ afin de former une construction d'amortissement des vibrations à couche libre.

13. Un procédé selon la revendication 1, dans lequel ladite construction à couche viscoélastique est une construction à couche contrainte, comportant:

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- a. le revêtement d'une couche amovible au moyen d'une couche d'une composition de résine époxy renfermant du fluorène;
 - b. la réticulation de ladite composition en une couche viscoélastique;
 - c. le transfert de ladite couche viscoélastique à partir dudit revêtement amovible sur un substrat, de façon à faire adhérer ladite couche viscoélastique sur ledit substrat afin de réaliser ladite construction d'amortissement des vibrations à couche contrainte; et
 - d. le montage solide de ladite construction d'amortissement des vibrations à couche contrainte sur un article vibrant qui doit être amorti.
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14. Un procédé selon la revendication 1, dans lequel ladite construction à couche viscoélastique est une construction à couche contrainte comportant:

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- a. le revêtement d'un substrat au moyen d'une couche d'une composition de résine epoxy;
 - b. la réticulation de la composition en une couche viscoélastique, in situ, sur un substrat afin de réaliser la construction d'amortissement des vibrations à couche contrainte; et
 - c. le montage solide de la construction d'amortissement des vibrations à couche contrainte sur un article vibrant devant être amorti.
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