HU000035007T2

(19) **HU**

(11) Lajstromszám: **E 035 007**

(13) **T2**

MAGYARORSZÁG
Szellemi Tulajdon Nemzeti Hivatala

EURÓPAI SZABADALOM
SZÖVEGÉNEK FORDÍTÁSA

(21) Magyar ügyszám: **E 13 792877**
(22) A bejelentés napja: **2013. 11. 12.**

(96) Az európai bejelentés bejelentési száma:
EP 20130792877
(97) Az európai bejelentés közzétételi adatai:
EP 2922900 A2 **2014. 05. 30.**
(97) Az európai szabadalom megadásának meghirdetési adatai:
EP 2922900 B1 **2017. 08. 09.**

(51) Int. Cl.:

C08J 9/00	*(2006.01)*
C08J 9/10	*(2006.01)*
C08L 33/04	*(2006.01)*
C08L 9/00	*(2006.01)*
C08L 23/08	*(2006.01)*
C08L 31/04	*(2006.01)*
B29C 44/18	*(2006.01)*
C08K 5/14	*(2006.01)*

(86) A nemzetközi (PCT) bejelentési szám:
PCT/EP 13/073537
(87) A nemzetközi közzétételi szám:
WO 14079723

(30) Elsőbbségi adatok:
102012221192 **2012. 11. 20.** **DE**

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(54) **Termikusan expandálható készítmények**

Thermally Expandable Formulations

[2] The present application relates to a thermally expandable preparation, comprising a specific peroxidically crosslinkable binary copolymer, a polymer based on one or more diene monomers, and a specific terpolymer, to baffle parts for sealing cavities which comprise this preparation, to a method for producing such baffle parts, and to a method for sealing cavities in components using such baffle parts.

[3] Modern automobiles and automobile parts have a plurality of cavities, which must be sealed to prevent the ingress of moisture and contaminants, as this can lead to corrosion on the corresponding body parts from within. This applies in particular to a modern integral body construction, in which a heavy frame construction is replaced with lightweight, structurally stable frame structures made of prefabricated hollow profiles. This kind of construction has a number of system-inherent cavities, which must be sealed to prevent the ingress of moisture and contaminants. Such sealing furthermore is used to avoid the transmission of airborne sound in such cavities and thereby reduce unpleasant vehicle running and wind noise, so as to thus increase the driving comfort in the vehicle.

[4] Frame and body parts containing such cavities can be prefabricated from half-shell elements, for example, which are joined at a later time by way of welding, riveting, clinching and/or bonding to form the closed hollow profile. Such a design consequently makes the cavity easily accessible at an early stage of construction of the vehicle body so that, during this phase of the body in white, sealing and sound insulating baffle parts can be affixed by mechanical mounting, by insertion into appropriate retaining devices, boreholes or by welding. Furthermore, such hollow profiles can be made of steel, aluminum or polymer materials using an extrusion process, by way of hydroforming, by way of die casting, or by way of drawing methods. The resulting cavities can then only still be accessed through the cross-sectional openings at the ends of these profiles.

[5] Baffle parts that cause a sealing and/or acoustic effect in such cavities are frequently referred to as pillar fillers, baffles or acoustic baffles. They are generally made entirely of thermally expandable molded bodies or of molded bodies that comprise a carrier and polymeric preparations expandable in the peripheral region.

[6] These baffle parts are secured in the body in white to the open structures by way of mounting, clipping, bolting or welding. After the structures in the body in white have been closed and further pretreatments of the body have been carried out, the process heat from the furnaces for curing the cathodic dip coating is used to trigger the expansion of the expandable portion of the baffle part, so as to seal the cross-section of the cavity.

[7] Both the design and geometry of such baffle parts, and the composition of the thermally expandable preparations are parameters that have been further optimized of late. Usually, baffle parts are used which are composed of a thermally expandable compound and a carrier material and which, in terms of the geometry thereof, are individually adapted to the hollow body to be damped.

[8] Since the production of two-component baffle parts from a carrier material and an expandable compound is very complex, there has been a need for quite some time to develop baffle parts that, even in the absence of a carrier structure, reliably seal the cavity without running off during curing. At the same time, the need exists to provide foams having high expansion rates, which can be used to shape universally usable baffle parts, ideally using no carrier material, which due to the high expansion are able to reliably seal cavities having a variety of geometries.

[9] For example, WO-A1-2001/30906 relates to self-supporting thermally expandable compounds and proposes that the compounds, prior to expansion, comprise at least one modified polyethylene, at least one hydrazide blowing agent, at least one hydrocarbon resin, and at least one sulfur-containing curing agent. The agent according to this document is characterized by expansion rates of up to 100%.

[10] In WO-A1-2008/034755 it is furthermore proposed to use thermally curable compounds having high expansion rates made of at least one anhydride-functionalized thermoplastic, an amine-functionalized latent curing agent and at least one latent blowing agent, which are preferably used together with a carrier material.

[11] It was the object of the present invention to provide thermally expandable compounds that do not require a carrier, yet have high expansion rates, so that the baffle parts produced from these compounds can be used universally in a variety of body cavity structures.

[12] Furthermore, the compounds according to the invention must well-tolerate the demands they experience in the curing process used in the automotive industry, which usually takes place as the produced vehicle passes through the furnaces for curing the cathodic dip coating.

[13] The problem during such a curing process is that not all parts of the vehicle are heated to the same temperature in the curing furnace. For example, some areas may come closer to the heat source during curing, causing these to be exposed to higher temperatures ("over-baking").

[14] Other areas, in contrast, may be shielded against the heat for design-related reasons and may not reach the optimal curing temperature ("under-baking"). In particular, the areas in the structural cavities are frequently shielded by thick metal walls and, due to the mass surrounding them, are not heated to optimal temperatures, which is to say these areas reach maximum temperatures only briefly, if at all, which are often considerably below the optimal conditions. It is therefore advantageous if the thermally expandable preparations are designed such that they sufficiently expand over a wide temperature range, and also with varying curing times, and in particular do not collapse at higher temperatures.

[15] The agents from the prior art have previously not been able to completely satisfy all the requirements with regard to such baffle parts.

[16] It has now been found that thermally expandable preparations that comprise a specific peroxidically crosslinkable binary copolymer, a polymer based on one or more diene monomers, and additionally a specific terpolymer, meet the requirements that are imposed on such thermally expandable preparations to a high degree. The preparations according to the invention have high durability during heating of the material necessary for curing/expansion and thus also allow high degrees of expansion to be achieved, if necessary even while dispensing with carrier material whatsoever, without the preparations sliding off the introduction site or slipping off downwardly under the influence of gravity.

[17] A first subject matter of the invention is thus thermally expandable preparations, comprising, based on the total weight of the thermally expandable composition,

(a) at least 40 wt.% of at least one peroxidically crosslinkable copolymer containing at least one monomer unit, selected from vinyl acetate, (meth)acrylic acids and the derivatives thereof, wherein the binary copolymer has a melt flow index of no more than 3 g/10 min, which is determined in accordance with DIN EN ISO 1133 using a test load of 2.16 kg and a test temperature of 190°C;

(b) 0.2 to 2 wt.% of at least one peroxide;

(c) 5 to 18 wt.% of at least one chemical blowing agent; and

(d) 2 to 20 wt.% of at least one polymer based on one or more diene monomers, which has a melt flow index of no less than 2 g/10 min, which is determined in accordance with DIN EN ISO 1133 using a test load of 2.16 kg and a test temperature of 150°C; and

(e) at least one terpolymer based on at least one first monomer, selected from the monounsaturated or polyunsaturated hydrocarbons, and at least one second monomer, selected from the (meth)acrylic acids and the derivatives thereof.

[18] A first component essential to the invention is the peroxidically crosslinkable binary copolymer, comprising at least one monomer unit selected from vinyl acetate, (meth)acrylic acids and the derivatives thereof, having a melt flow index of no more than 3 g/10 min. A person skilled in the art refers to polymers as being "peroxidically crosslinkable" if a hydrogen atom can be abstracted from the main chain or a side chain through the action of a radical starter, whereby a radical remains, which attacks other polymer chains in a second reaction step. According to the invention, "binary copolymers" shall be understood to mean all copolymers that result from a polymerization reaction of two different monomers. According to the invention, this shall, of course, also cover copolymers in which further monomer are incorporated into the polymer chain, for example due to decomposition reactions or impurities, in such low amounts that these do not influence the properties of the binary copolymer.

[19] The peroxidically crosslinkable binary copolymer according to the invention comprises at least one monomer unit selected from vinyl acetate, (meth)acrylic acids and the derivatives thereof. As usual, the prefix "(meth)" used before "acrylate" denotes that these monomers can be either acrylic acids and/or the derivatives thereof or methacrylic acids and/or the derivatives thereof. Particularly preferred monomer units of this group are vinyl acetate, butyl acrylate, methyl acrylate, ethyl acrylate, and 2-ethylhexyl acrylate. According to the invention, vinyl acetate is a particularly preferred representative of this group.

[20] The second monomer of the binary copolymer (a) according to the invention is preferably selected from the alkenes. Ethylene is a particularly preferred second monomer of the binary copolymer (a) within the meaning of the present invention.

[21] In a first preferred embodiment, the at least one peroxidically crosslinkable binary copolymer is selected from ethylene-vinyl acetate copolymers, functionalized ethylene-vinyl acetate copolymers, ethylene-butyl acrylate copolymers, functionalized ethylene-butyl acrylate copolymers, ethylene-methyl acrylate copolymers, ethylene-ethyl acrylate copolymers, ethylene-(meth)acrylic acid copolymers and ethylene-2-ethylhexyl acrylate copolymers.

[22] According to the invention, a "functionalized copolymer" shall be understood to mean a copolymer that is provided with additional hydroxide groups, carboxyl groups, anhydride groups, acrylate groups and/or glycidyl methacrylate groups, preferably at the ends of the chain.

[23] Ethylene-vinyl acetate copolymers, ethylene-butyl acrylate copolymers and the functionalized derivatives thereof are particularly advantageous within the meaning of the present invention. Ethylene-vinyl acetate copolymers, and in particular the representatives that comprise no functional groups can be especially particularly preferred according to the invention.

[24] Furthermore, the peroxidically crosslinkable binary copolymers according to the invention are characterized by a melt flow index of no more than 3 g/10 min. According to the invention, the melt flow index of the peroxidically crosslinkable polymers is determined by way of a melt flow meter, wherein the polymer is fused at 190°C in a heatable cylinder and pressed through a defined standard nozzle (DIN EN ISO 1133) at a pressure created by the applied load (2.16 kg). The exiting compound is ascertained as a function of the time.

[25] Peroxidically crosslinkable polymers, and in particular an optionally functionalized ethylene-vinyl acetate copolymer, having a melt flow index of 0.05 g to 2.5 g/10 min, and in particular of 0.1 g to 2.0 g/10 min, are particularly preferred according to the invention.

[26] Thermally expandable preparations that comprise at least one ethylene-vinyl acetate copolymer having a vinyl acetate content of 9 to 22 wt.%, in particular of 15 to 20 wt %, and most particularly of 17.5 to 19 wt.%, based on the total weight of the binary copolymer, are particularly preferred according to the invention.

[27] According to the invention, the thermally expandable preparations preferably comprise at least 40 wt.% of at least one or more of the peroxidically crosslinkable binary copolymers (a) according to the invention. Thermally expandable preparations that comprise 50 to 80 wt.%, and in particular 58 to 62 wt.%, of at least one or more of the peroxidically crosslinkable binary copolymers (a), in each case based on the total weight of the thermally expandable preparation, are particularly preferred.

[28] The thermally expandable preparations according to the invention comprise at least one peroxide as a second component essential to the invention. In particular, the organic peroxides, such as ketone peroxides, diacyl peroxides, peresters, perketals and hydroperoxides, are preferred according to the invention. Particularly preferred are, for example, cumyl hydroperoxide, t- butyl peroxide, bis(tert-butylperoxy)diisopropylbenzene, di(tert-butylperoxyisopropyl)benzene, dicumyl peroxide, t-butylperoxybenzoate, dialkyl peroxydicarbonate, diperoxy ketals (such as 1,1-di-tert-butylperoxy-3,3,5-trimethylcyclohexane), ketone peroxides (such as methyl ethyl ketone peroxides), and 4,4-di-tert-butylperoxy-n-butyl valerate.

[29] According to the invention, particularly preferred peroxides are those sold commercially, for example, by the companies Akzo Nobel and Pergan GmbH, such as 3,3,5,7,7-pentamethyl-1,2,4-trioxepane, 2,5-dimethyl-2,5-di(tert-butylperoxy)hex-3-in, di-tert-butyl peroxide, 2,5-dimethyl-2,5-di(tert-butylperoxy)hexane, tert-butylcumyl peroxide, di(tert-butylperoxyisopropyl)benzene, dicumyl peroxide, butyl-4,4-di(tert-butylperoxi)valerate, tert-butylperoxy-2-ethylhexyl carbonate, 1,1-di-(tert-

butylperoxy)-3,3,5-trimethylcyclohexane, tert-butyl peroxybenzoate, di-(4-methylbenzoyl)peroxide and dibenzoyl peroxide.

[30] It has furthermore proven advantageous according to the invention if the peroxides used are substantially inert at room temperature and only activated upon heating to higher temperatures (for example, upon heating to temperatures between 130°C and 240°C). It is particularly advantageous according to the invention when the peroxide used has a half life of more than 60 minutes at 65°C, which is to say that, after the thermally expandable preparation containing the peroxide has been heated to 65°C for 60 minutes, less than half of the peroxide used has decomposed. According to the invention, peroxides that, at 115°C, have a half life of 60 minutes can be particularly preferred.

[31] It may be particularly preferred according to the invention to use di(tert-butylperoxyisopropyl)benzene as peroxide; this is commercially available from Akzo Nobel, for example, under the trade names Perkadox® 14-40 B-PD or Perkadox® 14-40 K PD, or from Pergan under the trade names Peroxan® BIB 40 GS or Peroxan® BIB 40 P.

[32] In a further form according to the invention, it may likewise be preferred to use dicumyl peroxide, as it is sold commercially by Akzo Nobel, for example, under the trade names Perkadox® BC 40 K PD, Perkadox® BC 40BGR DD or Perkadox® BC 40 B PD, or by Pergan under the trade names Peroxan® DC 40 GS, Peroxan® DC 40 P or Peroxan® DC 40 PK. The use of dicumyl peroxide can be especially particularly preferred according to the invention.

[33] It is furthermore advantageous according to the invention if the at least one peroxide is, or the peroxides are, used in a form applied onto a solid inert carrier, such as calcium carbonate and/or silica and/or kaolin.

[34] The at least one peroxide is, or the peroxides are, present in the thermally expandable preparations according to the invention in an amount of 0.2 to 2 wt.%, preferably in an amount of 0.3 to 1 wt.%, and in particular in an amount of 0.4 to 0.6 wt.%, in each case determined as the active substance content of peroxide based on the total weight of the thermally expandable preparation.

[35] The thermally expandable preparations comprise at least one chemical blowing agent as a third component essential to the invention.

[36] A chemical blowing agent according to the invention shall be understood to mean compounds that decompose under the action of heat, releasing gases in the process.

[37] Examples of suitable chemical blowing agents are azo compounds, hydrazide compounds, nitroso compounds and carbazide compounds, such as azobisisobutyronitrile, azodicarbonamide (ADCA), di-nitroso-pentamethylene tetramine, 4,4'-oxybis(benzenesulfonic acid hydrazide) (OBSH), azocyclohexyl nitrile, azodiaminobenzene, benzene-1,3-sulfonyl hydrazide, calcium azide, 4,4'-diphenyldisulfonyl azide, diphenyl-sulfon-3,3'-disulfohydrazide, benzene-1,3-disulfohydrazide, trihydrazinotriazine, p-toluenesulfonyl hydrazide and p-toluenesulfonyl semicarbazide.

[38] The use of azodicarbonamide and/or sulfonic acid hydrazide has proven to be preferred according to the invention. Preferred sulfonic acid hydrazides are, in particular, 4,4'-oxybis(benzenesulfonic acid hydrazide) (OBSH), benzene-1 ,3-sulfonic acid hydrazide and 4-methylbenzene sulfonic acid hydrazide. Azodicarbonamide is an especially particularly preferred chemical blowing agent according to the invention.

[39] It may be preferred according to the invention if the thermally expandable preparations comprise a first blowing agent, which is already activated below 140°C, and a second blowing agent, which is only activated at temperatures above 160°C.

[40] According to the invention, preparations that have proven advantageous are those having a content of chemical blowing agents of 5 to 18 wt.%, in particular of 7 to 15 wt.%, and most particularly of 9 to 13 wt.%, in each case based on the total weight of the thermally expandable preparation.

[41] The chemical blowing agents according to the invention can advantageously be used in combination with activators and/or accelerators, such as zinc compounds (for example, zinc oxide, zinc stearate, zinc ditoluenesulfinate, zinc dibenzenesulfinate), magnesium oxide and/or (modified) ureas. The zinc compounds, and zinc oxide in particular, are particularly preferred according to the invention.

[42] According to the invention, it does not matter significantly whether the blowing agents are already used in activated form, or whether the thermally expandable preparations comprise an appropriate activator and/or accelerator, such as zinc oxide, in addition to the blowing agent.

[43] It has proven to be particularly advantageous when the thermally expandable preparations according to the invention comprise the activators and/or accelerators, in particular the zinc compounds, and most particularly the zinc oxide, in an amount of 0 to 15 wt.%, in particular of 0.2 to 5 wt.%, and especially particularly preferably of 1 to 3 wt.%, in each case based on the total weight of the thermally expandable preparation.

[44] Furthermore, preparations that have a content of already activated azodicarbonamide of 5 to 18 wt.%, in particular of 7 to 16 wt.%, and most particularly of 9 to 13 wt.%, in each case based on the total weight of the thermally expandable preparation, have proven advantageous, wherein the activated azodicarbonamide used has a content of activators of 1 to 10 wt.%, based on the amount of activated azodicarbonamide.

[45] The thermally expandable preparations comprise at least one polymer based on one or more diene monomers as a fourth ingredient essential to the invention.

[46] Although, in principle, no restrictions whatsoever exist with respect to the diene monomers, it has proven advantageous according to the invention when a polymer based on at least one alkadiene monomer is used. Homopolymers based on a diene monomer can be particularly preferred polymers (d) according to the invention.

[47] Although, in general, the use of non-functionalized polymers (d) is preferred, the polymers (d) can, in exceptions, certainly also be functionalized with additional hydroxide groups, carboxyl groups, anhydride groups, acrylate groups and/or glycidyl methacrylate groups, preferably at the ends of the chains.

[48] Diene monomers that are particularly preferred according to the invention are 1,2-butadiene, 1,3-butadiene and isoprene. 1,3-butadiene and isoprene are especially particularly preferred diene monomers according to the invention.

[49] Furthermore, polymers (d) having an average molar mass of at least 30,000 g/mol have proven to be preferred according to the invention. Polymers (d) having an average molar mass of at least 50,000 g/mol may be particularly preferred according to the invention. Average molar mass of polymers in the present context shall be understood to mean the weight average molecular weight (M_w), which can be determined by way of gel permeation chromatography (GPC) using polystyrene as the standard.

[50] Particularly advantageous properties were yielded when the polymer (d) is selected from the group consisting of the polybutadiene homopolymers, the polyisoprene homopolymers, and butadiene-isoprene copolymers. The preparations according to the invention that comprise these polymers (d) are characterized by good injection behavior during injection molding, good durability, and low run-off during curing.

[51] It is furthermore preferred according to the invention if the polymer (d) comprises 1,3-butadiene as a monomer ingredient.

[52] Moreover, polybutadienes based on 1,3-butadiene and having at least 90 mol% 1,2-linkages are preferred according to the invention. Such polybutadienes are also referred to as 1,2-polybutadienes according to the invention.

[53] It may likewise be preferred if the polymer (d) has a syndiotactic structure. Syndiotactic 1,2-polybutadiene is an especially particularly preferred polymer (d) in the meaning of the present invention.

[54] It is furthermore particularly preferred according to the invention if component (d) is solid at room temperature. According to the invention, polymers are referred to as being "solid" when the geometry of these polymers does not deform under the influence of gravity within 1 hour, and in particular within 24 hours, at the indicated temperature.

[55] According to the invention, the polymers based on one or more diene monomers used are those that have a melt flow index of at least 2 g/10 min, in particular of 2 to 10 g/10 min, and most particularly of 3 to 7 g/10 min. According to the invention, the melt flow index of component (d) is determined by way of a melt flow meter, wherein the polymer is fused at 150°C in a heatable cylinder and pressed through a defined standard nozzle (DIN EN ISO 1133) at a pressure created by the applied load (2.16 kg). The exiting compound is ascertained as a function of the time.

[56] According to the invention, preparations that have proven advantageous are those having a content of polymers based on one or more diene monomers of 2 to 20 wt.%, in particular of 3 to 20 wt.%, and most particularly of 4 to 7 wt.%, in each case based on the total weight of the thermally expandable preparation.

[57] As the fifth component essential to the invention, the thermally expandable preparations comprise at least one terpolymer based on at least one first monomer, selected from the monounsaturated or polyunsaturated hydrocarbons, and on at least one second monomer, selected from the (meth)acrylic acids and the derivatives thereof.

[58] It has proven to be preferred according to the invention if the first monomer unit of the terpolymer is a monounsaturated or polyunsaturated acyclic hydrocarbon; alkenes and dienes are particularly preferred representatives of this group; the monomer units ethylene, propylene, 1,2-butadiene, 1,3-butadiene and isoprene are especially particularly preferred representatives of this group according to the invention.

[59] The second comonomer of the terpolymer is selected from (meth)acrylic acid and the derivatives thereof. As usual, the prefix "(meth)" before "acrylate" denotes that these monomers can be either acrylic acids and/or acrylates, and methacrylic acids and/or the methacrylates. If the terpolymer according to the invention contains acrylates and/or methacrylates, the alcohol component of the ester is preferably selected from those containing 1 to 6 carbon atoms. In particular, methyl esters, ethyl esters and butyl esters may be used.

[60] In a preferred embodiment of the present invention, the third comonomer of component (e) is selected from the group consisting of styrene, glycidyl (meth)acrylates and maleic anhydride.

[61] According to the invention, glycidyl (meth)acrylicates shall be understood to mean the esters of acrylic acid or methacrylic acid with glycidol (2,3-epoxypropan-1-ol).

[62] Particularly good runoff behavior was achieved according to the invention when the following are used as the terpolymer (e):

- styrene-butadiene-(meth)acrylate acids;
- styrene-butadiene-(meth)acrylates;
- ethylene(meth)acrylates-glycidyl(meth)acrylates and/or
- ethylene-(meth)acrylate-maleic anhydrides, such as in particular ethylene-ethylacrylate-maleic anhydride and ethylene-butylacrylate-maleic anhydride.

[63] While according to the invention component (e) is defined as a terpolymer, the invention of course also covers copolymers that comprise further monomers, for example from decomposition reactions or impurities, in such low amounts that these do not influence the properties of the terpolymers according to the invention.

[64] The use of the terpolymers according to the invention in the preparations according to the invention enables better durability of the preparations during heating of the material necessary for curing/expansion. Furthermore, surprisingly it has been found that the use of these terpolymers allows uniform expansion even at different temperatures, which is to say that the degree of expansion of preparations containing these terpolymers varies less drastically than that of traditional preparations under under-baking, ideal-baking and over-baking conditions.

[65] In addition to the components essential to the invention, the thermally expandable preparations in a special embodiment may contain at least one low-molecular-weight multifunctional acrylate.

[66] According to the invention, a "low-molecular-weight multifunctional acrylate" shall be understood to mean a compound that comprises at least two acrylate groups and has a molecular weight of less than 2400 g/mol, and preferably less than 800 g/mol.

[67] According to the invention, in particular compounds comprising two, three or more acrylate groups per molecule have proven to be advantageous.

[68] Preferred difunctional acrylates are ethylene glycol dimethacrylate, diethylene glycol dimethacrylate, triethylene glycol dimethacrylate, triethylene glycol diacrylate, tripropylene glycol dimethacrylate, 1,4-butanediol dimethacrylate, 1,3-butylene glycol dimethacrylate, 1,3-butanediol dimethacrylate, tricyclodecane dimethanol dimethacrylate, 1,10-dodecanediol dimethacrylate, 1,6-hexanediol dimethacrylate, 2-methyl-1,8-octanediol dimethacrylate, 1,9-nonanediol dimethacrylate, neopentyl glycol dimethacrylate and polybutylene glycol dimethacrylate.

[69] Preferred low-molecular-weight acrylates comprising three or more acrylate groups are glycerol triacrylate, dipentaerythritol hexaacrylate, pentaerythritol triacrylate (TMM), tetramethylolmethane tetraacrylate (TMMT), trimethylolpropane triacrylate (TMPTA), pentaerythritol tetraacrylate, di(trimethylolpropane) tetraacrylate (TMPA), pentaerythritol tetraacrylate, trimethylolpropane trimethacrylate (TMPTMA), tri(2-acryloxyethyl) isocyanurate and tri(2-methacryloxyethyl) trimellitate, and the ethoxylated and propoxylated derivatives thereof, containing a maximum of 35 EO units and/or a maximum of 20 PO units

[70] Thermally expandable preparations comprising a low-molecular-weight multifunctional acrylate selected from triethylene glycol diacrylate, triethylene glycol dimethacrylate, trimethylolpropane triacrylate (TMPTA) and trimethylolpropane trimethacrylate (TMPTMA), pentaerythritol triacrylate (TMM), tetramethylolmethane tetraacrylate (TMMT), pentaerythritol trimethacrylate, di(trimethylolpropane) tetraacrylate (TMPA) and pentaerythritol tetraacrylate are especially particularly preferred according to the invention.

[71] According to the invention, it has proven to be particularly advantageous if the thermally expandable preparations comprise at least one low-molecular-weight multifunctional acrylate selected from triethylene glycol diacrylate, trimethylolpropane triacrylate (TMPTA) and trimethylolpropane trimethacrylate (TMPTMA).

[72] The low-molecular-weight multifunctional acrylates are preferably present in the thermally expandable preparations in an amount of 0.2 to 2.5 wt.%, and in particular of 0.4 to 1.4 wt.%, in each case based on the total weight of the thermally expandable preparation.

[73] According to the invention, use of the low-molecular-weight multifunctional acrylates has proven to be particularly advantageous for the stability of the resulting foam if the thermally expandable preparations either comprise little terpolymer (e) or little peroxide. In particular in the case of preparations having a peroxide content of no more than 1.5 wt.% and/or a terpolymer content of no more than 3 wt.%, the addition of low-molecular-weight acrylates has proven to be particularly advantageous.

[74] In the course of the work underlying the present application, however, it was also possible to demonstrate that the thermally expandable preparations can be further optimized with regard to the behavior thereof at suboptimal curing temperatures if they are formulated so as to be substantially free of these low-molecular-weight multifunctional acrylates. Compositions containing less than 0.25 wt.%, and in particular less than 0.15 wt.%, low-molecular-weight multifunctional acrylates are referred to as being "substantially free of these low-molecular-weight multifunctional acrylates." Thermally expandable compositions formulated so as to be free of low-molecular-weight multifunctional acrylates may be especially particularly preferred according to the invention.

[75] In a further embodiment of the present invention, it may be preferred if the thermally expandable compositions furthermore contain at least one hydrocarbon resin.

[76] According to the invention, "hydrocarbon resins" shall be understood to mean thermoplastic polymers according to the invention which can be obtained from petroleum fractions and have an average molar mass of no more than 2500 g/mol. Hydrocarbon resins having an average molar mass of no more than 2,000 g/mol may be particularly preferred according to the invention. The average molar mass of polymers within the scope of the present application shall generally be understood to mean the weight average molecular weight. Within the scope of the present invention, the weight average molecular weight (M_w) can be determined by way of gel permeation chromatography (GPC) using polystyrene as the standard.

[77] The hydrocarbon resins may be completely aliphatic or completely aromatic, or they may have aliphatic and aromatic structures. Furthermore, these may be aromatically modified aliphatic resins. In any case, compatibility with the polymer matrix is essential. Commercial products such as Escorez® 1102, Escorez® 2173, Escorez® 2184, Escorez® 2101, Escorez 2105, Novares® TK, Novares® TL 100, Novares® TV, Novares® TA, Novares® TP, Novares® TR, Novares® TS, Novares® TW, Necires® LF 220 and Nevtac® 10 may be used for this purpose.

[78] Hydrocarbon resins having a softening point of > 10°C, preferably having a softening point of > 40°C, and in particular having a softening point of > 70°C are particularly preferred according to the invention.

[79] The hydrocarbon resins are preferably present in the thermally expandable preparations in an amount of 0.2 to 25 wt.%, in particular of 5 to 20 wt.%, and especially particularly preferably of 8 to 15 wt.%, in each case based on the total weight of the thermally expandable preparation.

[80] The thermally expandable preparations according to the invention are characterized in particular by not sliding off the introduction site under the influence of gravity during the curing process, even at high expansion rates, or by not slipping downwardly during expansion. Rather, they expand at the site at which they were introduced into the cavity and expand in the direction of the opposing walls of the cavity. It is therefore particularly preferred according to the invention if the thermally expandable preparations have a degree of expansion of at least 1000%, preferably of at least 1500%, and in particular of at least 2000%. The indicated degree of expansion therefore refers to the volume of the composition at room temperature before and after being heated for 30 minutes to an activation temperature of 170°C.

[81] In addition to the ingredients according to the invention, the thermally expandable compounds can also comprise further customary components, such as dyes, fillers and antioxidants

[82] Fillers that may be used include, for example, the various ground or precipitated chalks, calcium magnesium carbonates, talc, graphite, heavy spar, silicic acids or silica and in particular siliceous fillers, such as mica, for example in the form of chlorite, or siliceous fillers of the aluminum-magnesium-calcium silicate type, for example, wollastonite. Talc is a particular preferred filler.

[83] The fillers are preferably used in an amount of 0 to 16 wt.%, and in particular of 0.1 to 10 wt.%, in each case based on the weight of the total thermally expandable preparation.

[84] Color-imparting components, and in particular black dyes based on carbon blacks, are preferably present in the thermally expandable preparations according to the invention in an amount of 0 to 2 wt.%, in particular of 0.1 to 0.8 wt.%, and especially particularly preferably 0.15 to 0.4 wt.%, in each case based on the weight of the thermally expandable preparation.

[85] Sterically hindered phenols and/or sterically hindered thioethers and/or sterically hindered aromatic amines may be used as antioxidants or stabilizers, such as bis-(3,3-bis-4'-hydroxy-3-tert-butylphenyl)butanoic acid) glycol esters.

[86] Antioxidants or stabilizers are preferably present in the thermally expandable preparations according to the invention in an amount of 0 to 0.5 wt.%, and in particular of 0.1 to 0.4 wt.%, in each case based on the weight of the total thermally expandable preparation.

[87] The thermally expandable preparations according to the invention are preferably formulated so as to be solid at 22°C. According to the invention, a thermally expandable preparation is referred to as being "solid" when the geometry of this preparation does not deform under the influence of gravity within 1 hour, and in particular within 24 hours, at the indicated temperature.

[88] The thermally expandable preparations according to the invention can be produced by mixing the selected components in any arbitrary suitable mixer, such as a kneader, a double-Z kneader, an internal mixer, a twin-screw mixer, a continuous mixer or an extruder, and in particular a twin-screw extruder.

[89] Although it may be advantageous to heat the components slightly to facilitate achieving a homogeneous, uniform compound, care must be taken to ensure that temperatures capable of activating the peroxide and/or the blowing agent mixture are not reached. The resulting thermally expandable preparation can be shaped immediately after being produced, for example by way of blow molding, pelletizing, injection molding methods, compression molding methods, stamping methods or extrusion.

[90] Although it may be preferred according to the invention to prepare the entire thermally expandable preparation, then extrude and store it in pellet form, for example, until the baffle parts are produced, it has also proven to be advantageous according to the invention to formulate the thermally expandable preparation as a two-component agent up to the thermoforming stage.

[91] These two-component agents preferably comprise a first component, which has a low content of the total amount of peroxidically crosslinkable polymer and all the remaining components. This first component can be prepared and stored separately. This separation of the overall preparation into two components allows a cost-optimized production of the baffle parts since all the critical ingredients are present in the first formulation, which has a lower weight than the final thermally expandable preparation. Preferably a maximum of 20% of the total amount of the peroxidically crosslinkable polymer is present in the first component. The second component then contains the remaining amount of the polymer and optionally further auxiliary substances and additives. It may be preferred according to the invention if the second component contains only the remaining amount of the polymer.

[92] Prior to production of the baffle parts, the two-component agent can then be mixed in an injection molding machine comprising a twin screw and injected into the desired mold. However, it is also possible according to the invention to mix the two-component agent first with the complete thermally expandable preparation, to pelletize it, and then to inject the complete pelletized preparation into the mold in a separate step.

[93] The expansion of the thermally expandable preparation takes place by way of heating, wherein the preparation is heated to a certain temperature for a certain period of time sufficient to induce activation of the blowing agent and of the peroxide.

[94] Depending on the makeup of the preparation and the conditions of the production line, such temperatures are usually in the range of 130°C to 240°C, and preferably 150°C to 200°C, with a residence time of 10 to 90 minutes, and preferably 5 to 60 minutes.

[95] In vehicle construction, it is particularly advantageous for the expansion of the preparations according to the invention to take place as the vehicle passes through the furnace for curing the cathodic dip coating, so that a separate heating step can be dispensed with.

[96] The thermally expandable preparations of the present invention can be used in a wide range of sealing and adhesive applications, for example in the field of baffle parts for sealing cavities in vehicles. However, use as a lining adhesive, for example in the door or roof area, is also conceivable. For such an intended purpose, the thermally expandable preparations according to the invention can be applied by way of direct extrusion. However, the preparations can also be brought to the site of application in the extruded form and pressed and partially fused there by heating the steel. As a third alternative, it is also conceivable to apply the preparations as a co-extrudate. In this embodiment, according to the invention a second tacky preparation is applied in a thin layer beneath the actual non-tacky molded body made of the thermally expandable preparation according to the invention. Within the scope of this embodiment, this second tacky layer is used to affix the molded body in the body in white.

[97] The thermally expandable preparations are thus particularly suitable for producing baffle parts used to seal cavities, which is to say for producing parts that are introduced into the cavities of vehicles, then expanded by heating while curing, so as to thereby seal the cavity as completely as possible.

[98] A second subject matter of the present invention is thus a baffle part for sealing cavities of a component having a shape that is adapted to the cavity and comprising a thermally expandable preparation according to the invention.

[99] A "shape adapted to the cavity" according to the invention shall be understood to refer to all geometries of baffle parts which ensure complete sealing of the cavity after expansion. The shape of the baffle part can individually replicate the shape of the cavity and may have corresponding tips and/or rounded regions; in the case of the thermally expandable compositions according to the invention having high degrees of expansion, however, it may also suffice to introduce a suitably large amount in a variable shape, for example in the form of a bead or a strand of the material cut to length, into the cavity to ensure complete sealing of the cavity after expansion.

[100] Such baffle parts are usually produced from the thermally expandable preparations according to the invention by way of injection molding techniques. The thermally expandable preparations are heated to temperatures in the range of 70 to 90°C and then injected into a suitably designed mold.

[101] It is preferred according to the invention if the baffle parts comprise at least one fastening element, which allows anchoring of the baffle part in the cavity.

[102] In a particularly preferred embodiment of this subject matter of the present invention, the baffle parts are fabricated completely from the thermally expandable preparation. The preparation can thus be brought to the desired shape of the baffle part, for example by way of an injection molding process, by punching it out of a prefabricated panel or by extrusion through a die and subsequently cutting it to length. It is not necessary to use a carrier material in this embodiment.

[103] In this embodiment, fastening elements are an integral component of the baffle part, which is to say these are likewise fabricated from the thermally expandable preparation. Such fastening elements may take on the shape of an indentation and may thus contribute to the baffle part remaining in the intended location in the cavity.

[104] The fastening elements may thus be designed, for example, such that they can be introduced into an opening in the cavity, wherein they are preferably designed such that they cannot be removed from the opening again (for example, through the use of hooks or suitable elevations). It is particularly preferred if the fastening elements are fabricated from the thermally expandable preparation since this will also completely seal these openings during the course of the expansion due to heating.

[105] In another embodiment of this subject matter of the present invention, only the main component of the baffle part is fabricated from the thermally expandable preparation. In addition to the expandable main part, the baffle parts of this embodiment comprise fastening elements made

of another non-expandable materials such as metal or heat-resistant plastic. Thus, for example, a pin or a compressible plug may be anchored as a fastening element on an edge in the thermally expandable preparation, so as to be insertable into an opening in the cavity to be sealed.

[106] Although the thermally expandable compounds according to the invention allow baffle parts to be fabricated without a carrier material, these can nevertheless also be used in the traditional manner with a carrier. In this embodiment, the baffle part comprises a carrier to which the thermally expandable preparation is applied. In this embodiment, the carrier can be used to direct the expanding foam toward the walls of the cavity so as to prevent the foam from collapsing or deforming in another undesirable manner. The amount of thermally expandable preparation in the baffle part is preferably selected such that the foamed material completely fills the space between the baffle part and the cavity walls, and thereby seals the cavity and prevents the transmission of noise.

[107] The carrier is preferably made of a thermoplastic material that is sufficiently fracture-resistant under normal usage conditions and has a melting point or softening point above the curing temperature of the component. The carrier is preferably made of a plurality of polymeric materials such as polyesters, aromatic polyethers, polyether ketones and in particular polyamides, such as Nylon 66. In addition to the polymeric ingredients, the carrier material may contain further additives and fillers, such as dye and/or reinforcing fibers. Alternatively, the carrier may also be fabricated from metal, for example steel or aluminum.

[108] The thermally expandable preparation can be produced together with the carrier using traditional methods. In particular, injection molding methods in which the two components are injected are particularly advantageous. However, it is also preferred according to the invention if the carrier material is injected in a separate step and the thermally expandable composition is applied to the carrier only thereafter in a separate step, optionally even by means of a separate machine.

[109] The baffle parts according to the invention may be used in all products that have cavities. In addition to vehicles, these also include, for example, airplanes, rail vehicles, household appliances, furniture, buildings, walls, partitions or boats.

[110] Another subject matter of the present invention is a method for sealing cavities of a component, wherein a baffle part according to the invention is introduced into the cavity and then heated to a temperature above 130°C, so that the thermally expandable preparation expands and seals the cavity.

[111] What was said above with respect to the other subject matter applies, *mutatis mutandis*, to the details of this subject matter of the present invention.

[112] Another subject matter of the present invention is the use of a baffle part according to the invention for sound insulating cavities in components and/or for sealing cavities in components against water and/or moisture.

[113] What was said above with respect to the other subject matter applies, *mutatis mutandis*, to the details of this subject matter of the present invention.

[114] Exemplary Embodiments

[115] *1 Production of the Formulations*

[116] In a kneading mixer, the raw materials Escorene® Ultra UL 00218 CC 3 and RB 810 were first mixed at 130°C in accordance with the specifications in Table 1 until a homogeneous compound was obtained. Thereafter, the remaining raw materials were successively added, wherein the mixture was cooled so that the mixture was not heated to temperatures above 99°C.

[117] *2 Determination of the Expansion*

[118] To determine the expansion, test specimens having the dimensions 240 mm × 240 mm × 6 mm were cut from the finished panels and then inserted into a circulating air oven, which was heated to the temperature listed in Table 1 (heating time approx. 7 to 10 min), and the test specimens were then left at this temperature for the period of time listed in Table 1. The expansion at 170°C corresponds to the ideal conditions, which are achieved as part of curing in vehicle construction. The expansion at 150°C simulates the under-baking conditions, while the expansion at 190°C simulates the over-baking conditions.

[119] The extent of the expansion was determined by means of the water displacement method according to the formula

$$\text{Expansion} = \frac{(m2-m1)}{m1} \times 100$$

m1 = mass of the test specimen in the original state, in deionized water
m2 = mass of the test specimen after baking, in deionized water.

[120] *3 Determination of the Runoff Behavior*

[121] To determine the runoff behavior, test specimens having the dimensions 10 mm × 10 mm × 4 mm were cut from finished panels and then fused on a horizontal oiled metal sheet (galvanized zinc, oiling with 3 g/m²) for 5 minutes at 100°C in a circulating air oven. After cooling, the metal sheet thus prepared was introduced vertically for 30 minutes at 175°C in a circulating air oven, so that the product would expand. After the metal sheet was removed from the oven and cooled, the runoff and/or slippage of the resulting foam was assessed in comparison with the starting position.

[122] *4 Formulations and Measurement Results*

[123] 4.1 Tabular overview

[124] The quantity information shall be understood to be provided in percent by weight, unless noted otherwise.

Table 1:

	VV1	VV2	VV3	VV4	E1	E2	E3
Elvax® 470A	71.1	69.8	73.9	71.4	-	59.4	58.9
Escorene® Ultra UL 00218 CC 3	-	-	-	-	59.4	-	-
RB 810	2.8	5.00	-	2.8	5.0	5.0	5.0
Novares® TL 100	10.5	-	10.8	10.4	10.4	10.4	10.5
Necires® LF 220	-	10.5	-	-	-	-	-
Zinc Oxide Activox® B	2.8	2.8	2.9	2.8	2.7	2.7	2.8
Monarch® 280	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Perkadox® BC 40BGR DD	0.6	0.6	0.6	1.3	1.3	1.3	1.3
Celogen® AZ 130	11.1	11.1	11.6	11.1	11.1	11.1	11.1
Lotader® AX 8900	-	-	-	-	9.9	9.9	10.0

Sartomer® SR 350	0.9	-	-	-	-	-	0.2
TOTAL	100	100	100	100	100	100	100
Expansion at 30 min @ 150°C [%]	1500-1600	1500-1600	1200-1300	1200-1300	1800-1900	1600-1700	1400-1500
Expansion at 30 min 170°C [%]	1400-1500	1400-1600	200-300	2000-2100	2500-2600	2100-2200	1950-2000
Expansion at 30 min @ 190°C [%]	500-600	750-800	200-300	800-900	2400-2500	1800-1900	1700-1800
Runoff behavior	no runoff, no slippage	heavy runoff, heavy slippage	heavy runoff	heavy runoff	no runoff, no slippage	no runoff, no slippage	no runoff, no slippage

[125] 4 2 List of Commercial Products Used

Celogen® AZ 130	Azodicarbonamide (Safic Alcan)
Elvax® 470A	Ethylene-vinyl acetate copolymer (approx. 18 wt.% vinyl acetate content in the copolymer, melting point 89°C, melt flow index 0 7 g/10 min at 190°C under a load of 2.16 kg) (DuPont)
Escorene® Ultra UL 00218 CC 3	Ethylene-vinyl acetate copolymer (approx. 18 wt.% vinyl acetate content in the copolymer, melting point 86°C, melt flow index 1.7 g/10 min at 190°C under a load of 2.16 kg) (Exxon Mobil)
Lotader® AX 8900	Terpolymer of ethylene, acrylate and glycidyl methacrylate with a random arrangement (acrylate content 24 wt.%, glycidyl methacrylate content 8 wt.%) (Arkema)
Monarch® 280	Carbon black (degree of purity at least 99%) (Cabot)
Necires® LF 220	Hydrocarbon resin, polymerization product of cycloaliphatic and alkylaromatic monomers (Rüttgers Chemicals)
Novares® TL 100	Hydrocarbon resin, polymerization product of unsaturated aromatic C_9 to C_{10} hydrocarbons (Rüttgers Chemicals)
Perkadox® BC 40BGR DD	Dicumyl peroxide on a chalk-silica carrier approx. 40 wt.% active substance content (Akzo Nobel)
RB 810	Syndiotactic 1,2-polybutadiene homopolymer (melt flow index 3 g/10 min at 150°C under a load of 2.16 kg; melting point 71°C) (Japan Synthetic Rubber)

Sartomer® SR 350	Trimethylolpropane trimethacrylate (Sartomer)
Zinc oxide Activox® B	Zinc oxide (degree of purity 99.9%) (NRC Nordmann Rassmann)

[126] 4.3 Evaluation

[127] The exact compositions of the various formulations and the results of the determination of the expansion behavior and of the runoff behavior under various baking conditions were summarized in Table 1.

[128] While preparations E1 to E3 according to the invention have expansion values of 1400 to 2600% under the examined conditions, in particular advantageous values of 1950 to 2600% under standard conditions and over-baking conditions, comparison formulations VV1 to VV4 show a considerably lower expansion volume of 200 to 2100%. In particular in the case of over-baking conditions, comparison formulations VV1 to VV4 only achieved maximum expansion values of 900%.

[129] In contrast, under all the baking conditions tested, the expansion values of all formulations E1 to E3 according to the invention remain at a consistently high level of more than 1400%, while comparison formulations VV1 to VV4 have much greater fluctuations; shrinkage of the comparison formulations can in particular be observed under over-baking conditions (30 min @ 190°C), which can result in leaks in the application field. Based on the consistently high expansion values of compositions E1 to E3 according to the invention, it is possible to seal cavities completely and reliably using these compositions.

[130] In addition, the results in Table 1 indicate that compositions E1 to E3 according to the invention do not run off, slip off or sag during baking. In particular in the vertical position, the foams remain stable during the baking process. In contrast, comparison formulations VV2 to VV4 show extensive runoff and slipping during baking so that, in a vertical position, a cavity cannot be filled entirely with these formulations. Even though VV1 likewise does not exhibit any runoff/slipping during baking, the expansion is inadequate, in particular under over-baking conditions, since the foam has the disadvantage of shrinking.

Termikusan expandálható készítmények

Szabadalmi igénypontok

1. Termikusan expandálható készítmény, amely a termikusan tágítható készítmény össztömegére vonatkoztatva

(a) legalább 40 tömeg %, legalább egy peroxidosan térhálósítható bináris kopolimert tartalmaz, amely a következőket tartalmazza

legalább egy monomeregység, amelyet a vinilacetát, (met)akrilsavak és ezek származékai közül választunk, ahol a bináris kopolimer folyási indexe legfeljebb 3g/10 perc, amelyet a DIN EN ISO 1133 szabványnak megfelelően, 2,16 kg próbaterhelés és 190 C° próbahőmérséklet mellett mértünk,

(b) legalább egy peroxid, 0,2 - 2 tömeg % mennyiségben,

(c) legalább egy vegyi hajtóanyag, 5 - 18 tömeg % mennyiségben, illetve

(d) legalább egy polimer, 2 - 20 tömeg % mennyiségben, amely polimer egy vagy több dimonomer bázisú, és folyási indexe legalább 2g/10 perc, amelyet a DIN EN ISO 1133 szabványnak megfelelően, 2,16 kg próbaterhelés és 150 C° próbahőmérséklet mellett mértünk, és

(e) legalább egy terpolimer, amely legalább egy első monomeren alapszik, amelyet az egyszeresen vagy többszörösen telítetlen szénhidrogének csoportjából választunk és legalább egy második monomeren alapszik, amelyet a (met)akrilsavak és származékaik közül választunk.

2. Az 1. igénypont szerinti termikusan expandálható készítmény, azzal jellemezve, hogy az (a) peroxidosan térhálósítható polimert az alábbiak közül választjuk: etilén-vinilacetát kopolimerek, funkcionalizált etilén-vinilacetát kopolimerek, etilén-butilakrilát kopolimerek, funkcionalizált etilén-butilakrilát kopolimerek, etilén-metakrilát kopolimerek, etilén-etilakrilát kopolimerek, etilén-(met)akrilsav-kopolimerek és etilén-2-etilhexilakrilát kopolimerek.

3. Az 1. vagy 2. igénypont szerinti készítmény, azzal jellemezve, hogy (a) komponensként egy előnyösen a bináris kopolimer teljes tömegére vonatkoztatott 9 - 22 tömeg % vinilacetát-részsel rendelkező etilén-vinilacetát kopolimert tartalmaz.

4. Az 1-3. igénypontok valamelyike szerinti termikusan expandálható készítmény, azzal jellemezve, hogy hajtóanyagként egy szulfonsavhidrazidot, és/vagy azodikarbonamidot tartalmaz.

5. Az 1-4. igénypontok valamelyike szerinti termikusan expandálható készítmény, azzal jellemezve, hogy a (d) polimert a polibutadién homopolimerek, a poliizoprénhomopolimerek, illetve a butadién-izoprénkopolimerek közül választjuk.

6. Az 1-5. igénypontok valamelyike szerinti termikusan expandálható készítmény, azzal jellemezve, hogy a (d) polimer szindiotaktikusan felépített.

7. Az 1-6. igénypontok valamelyike szerinti termikusan expandálható készítmény, azzal jellemezve, hogy az (e) terpolimer egy harmadik, a sztirol, glicidil(met)akrilsavészterek és a maleinsavanhidrid közül választott monomeregységet is tartalmaz.

8. Az 1-7. igénypontok valamelyike szerinti termikusan expandálható készítmény, azzal jellemezve, hogy továbbá legalább egy termoplasztikus polimert tartalmaz, amely kőolajfrakciókból nyerhető ki, és molekulatömege legfeljebb 2500 g/mól (szénhidrogéngyanta), amelyet gélpermeációs kromatográfiával (GPC), standardként polisztirolt alkalmazva határozunk meg.

9. Az 1-8. igénypontok valamelyike szerinti termikusan expandálható készítmény, azzal jellemezve, hogy expanziós foka legalább 1000 % a masszának egy 30 percen át 170 C°-os aktiválási hőmérsékletre történő melegítése előtt és után szobahőmérsékleten mért térfogatához viszonyítva.

10. Tömítő rész egy építőelem üregeinek lezárására, azzal jellemezve, hogy az üreghez illesztett formával rendelkezik, és egy az 1-9. igénypontok valamelyike szerinti termikusan expandálható készítményt tartalmaz.

11. A 10. igénypont szerinti tömítő rész, azzal jellemezve, hogy legalább egy rögzítő elemmel rendelkezik, amely lehetővé teszi az üregben történő rögzítést.

12. A 10. vagy 11. igénypont szerinti tömítő rész, azzal jellemezve, hogy nem rendelkezik hordozó szerkezettel.

13. Eljárás egy építőelem üregeinek lezárására, amelynek során egy a 10-12. igénypontok valamelyike szerinti tömítő részt az üregbe helyeztünk, és ezt követően 130 C° fölötti hőmérsékletre melegítjük, amelynek eredményeképpen a termikusan expandálható készítmény expandál, és tömíti az üreget.

14. A 10-12. igénypontok valamelyike szerinti tömítő rész alkalmazása építőelemek üregeinek akusztikus tömítésére és/vagy építőelemek üregeinek vízzel szembeni tömítésére.