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Siloxane-containing casting resin system.

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☐ Drawing(s) : paged to paged
☒ Abstract : paged 7 to paged

RTM
60p

5 Temperature- and colour-stable casting resins are required as a covering for optoelectronic components capable of SMT, for example for optoelectronic multichip arrays.

10 Transparent colour-stable casting resins are known, for example, from DE 26 42 465. They are preferably employed for sheathing LEDs and LEDs capable of SMT, so-called TOPLEDs. These resins are of only limited suitability for covering components of large surface area, such as, for example, for covering the abovementioned multichip arrays. Coverings of large surface area of the known resins show cracks and adhesion problems which occur, in particular, after temperature shock
15 treatment or after the solder heat required for soldering. These can impair the optical and/or electrical properties of the optoelectronic components.

20 The object of the present invention is therefore to provide a casting resin which is suitable for covering optoelectronic components, with which a heat-stable covering which is also colour-stable over a relatively long life can be produced, and which avoids the disadvantages of the casting resins known for optoelectronic components. In particular, the moulded material should
25 resist high continuous operating temperatures of 125°C and the exposure to heat during the soldering process.

30 This object is achieved according to the invention by a casting resin system according to Claim 1. Further embodiments of the invention, in particular a process for the preparation of the casting resin system and a preferred use, are to be found in the sub-claims.

35 Surprisingly, it has been found that the epoxy resin systems according to the invention modified with siloxane solve the abovementioned problem in an outstanding manner. The casting resins, or their A-components,

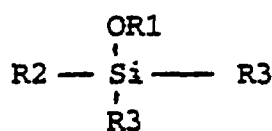
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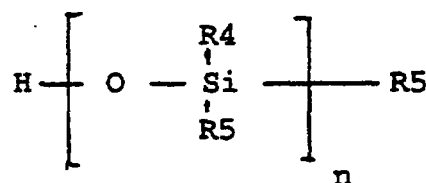
are water-clear and transparent. They can be stored at room temperature for several months without a substantial increase in their viscosity or without a reduction in the epoxide content, and can be processed as conventional epoxy casting resins. The chemistry and the reactivity of the epoxy resin is not adversely influenced by by [sic] the epoxyalkoxysiloxanes.

Although the moulded materials which can be obtained from the casting resin system according to the invention by curing are flexibilized due to the addition of the epoxyalkoxysiloxane, compared with the siloxane-free moulded materials, they do not have the disadvantages usually associated with this property. Thus, the glass transition temperature of the moulded materials is only insignificantly lower than that of a siloxane-free comparison resin. The modulus of elasticity also remains high, while the elastic properties are improved. The heat resistance, which manifests itself in particular in a lower rate of thermal degradation and in a higher decomposition temperature, is improved. The moulded materials here are clear and transparent. In the accelerated ageing process, they display a high colour stability and meet the required specifications.

A particularly advantageous casting resin system is obtained if the epoxysiloxane contained in the A-component to the extent of 5 to 95 % by weight is prepared by condensation of an epoxyalkoxysilane 1 with a silanol 2



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R1 is an alkyl radical having 1 to 6 C atoms or an aryl radical,

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R2 is a glycidylalkyl, epoxyalkyl or an epoxycycloalkyl radical, the two radicals

R3 independently of one another are OR1 or R2 or another alkyl or aryl radical,

5 R4 is alkyl or aryl, the radicals

R5 independently of one another are OH or R4 and

n is an integer, where $1 \leq n \leq 12$.

This reaction can be carried out in a simple one-pot reaction under normal conditions with respect to
10 atmosphere and pressure and with a minimum expenditure on the process to give reproducible siloxane condensation products in a very short time. The primary products are readily compatible and can easily be mixed in all proportions with commercially obtainable epoxides. Such
15 mixtures are stable to storage and can be crosslinked with the carboxylic anhydrides customary for casting resin technology.

The epoxyalkoxysilane 1 carries 1 to 3 alkoxy groups which are capable of condensation. The radical R1
20 can be chosen as desired, but is preferably an alkyl radical having 1 to 6 C atoms, since the reactivity of the group to be split off during the condensation decreases with increasing chain length of the alkyl radical. The most reactive starting compounds are thus
25 epoxymethoxysilanes 1. Since alcohol is split off during the condensation, the alkyl group R1 can also preferably be ethyl, because of the acceptability of ethyl alcohol.

Monomeric epoxyalkoxysilanes 1 are preferred, because of their easier availability, but the reaction is
30 in principle also possible with correspondingly longer-chain alkoxy siloxanes.

Longer-chain silanols 2 which contain the OH groups in the alpha-position, alpha- and omega-position or in the chain are relatively easily available and also
35 relatively inexpensive. The further organic group R4 bonded via SiC is not critical with respect to its choice and can be any desired alkyl or aryl radical. The index n, which determines the number of siloxane units, can be chosen freely between 1 and 12. Depending on the other



radicals, however, an increasing incompatibility of the condensation products (epoxyalkylsiloxanes) may result with increasing chain length, which may render their use in the casting resin system according to the invention more difficult or impossible.

The radical R2 containing epoxide groups in the starting compound 1 is bonded to silicon via a C atom and can otherwise be chosen freely. R2 can be a glycidylalkyl or an epoxyalkyl epoxyaryl or epoxycycloalkyl group, depending on the availability of the corresponding epoxyalkoxysilane. The corresponding glycidyloxy compounds which are obtained by reaction of correspondingly reactive compounds with epichlorohydrin are readily available.

A condensation catalyst may be necessary to assist the reaction, depending on the reactivity of the starting substances, which may be hindered electronically and sterically. There are no limitations on the catalyst in respect of the reaction itself, so that any desired condensation catalyst is suitable. A prerequisite is, however, that the catalyst does not influence the epoxide group and likewise does not impair the storage properties and the curing process of the casting resin system. An ideal catalyst therefore has neither a decidedly basic nor a decidedly acid reaction, and in the ideal case is neutral.

The reaction of the starting substances 1 and 2 can be carried out in solution or as a bulk reaction.

Preferred reaction temperatures are between 80 and 150°C, volatile reaction products preferably being blown out in an open reaction vessel by passing in a stream of inert gas. The storage life and stability both of the epoxyalkylsiloxane and of the casting resin system are increased in this way. The storage stability of the casting resin system can be increased by heating the A-component under an applied vacuum.

The condensation of 1 and 2 is preferably carried out under approximately stoichiometric conditions, so that approximately the same number of groups capable of



condensation are available in both reaction partners, that is to say hydroxide groups in the silanol 2 and alkoxy groups in 1.

5 The process for the preparation of the casting resin system according to the invention is described in more detail below with the aid of embodiment examples. Preparation of the siloxane-modified A-component:

3-Glycidylpropyltrimethoxysilane (GPT), which is already known as an adhesion promoter for epoxy resins and is admixed to customary epoxy resins in amounts of
10 about 0.25 per cent, is used as the alkoxy silane 1. α,ω -Propylphenylsilanediol (PPS) is chosen as the silanol 2.

57.8 g of PPS are added in portions to 11.8 g of
15 GPT (50 mmol) at 120°C in the course of 15 minutes. The amount of silanol is chosen such that amounts of OH groups equivalent to the methoxy functions are available for the condensation. The solid PPS dissolves, with evolution of methanol, and a highly viscous siloxane
20 condensate forms. During this procedure, the reaction mixture is stirred at 120°C for a total of 4 hours, while distilling off methanol and passing over nitrogen at the same time. This methanol-containing condensate comprises various siloxane products and is processed directly with
25 the desired amount of epoxy casting resin at 120°C to give a storage-stable siloxane-containing A-component.

For this, an epoxyalkylsiloxane (siloxane condensate) prepared as above is mixed in various mixing ratios with the cycloaliphatic epoxide CY 179 (3,4-epoxycyclo-
30 hexylmethyl 3',4'-epoxycyclohexanecarboxylate, Ciba-Geigy).

For example, the viscosity at 60°C and the epoxide value of an A-component comprising four parts of siloxane condensate and one part of CY 179 change only
35 slightly after storage at 60°C for 6 months. During this period, the viscosity rises from 120 to 160 mPas and the epoxide value drops from 0.581 to 0.574 mol/100 g.

The following table shows some properties of mixtures V1 to V4, which are compared with the known pure



cycloaliphatic epoxide in Experiment V0.

Characteristic data of the A-components

	V0	V1	V2	V3	V4
5 Epoxide value (mol/100g)	0.73	0.58	0.39	0.26	0.20
Condensate content (%)	0	20	50	70	80
10 Viscosity η (60°C) (mPas)	40	120	240	5,800	7,000
n_D (20°C)	1.498	1.505	1.513	1.518	1.519
Colour	water-clear, transparent	water-clear, transparent	water-clear, transparent	water-clear, transparent	water-clear, transparent

Production of a transparent moulded material

15 The content of cycloaliphatic epoxide determines the viscosity and the epoxide value and thus the processability of the A-component prepared by mixing, to which a stoichiometric amount of B-component is added, according to its epoxide content. This comprises a carboxylic acid anhydride, which can be partly esterified with an

20 alkyl alcohol. Suitable anhydrides are, for example, (methyl-)tetrahydrophthalic anhydride or (methyl-)hexahydrophthalic anhydride. Hexahydrophthalic anhydride which is mixed with 13 parts by weight of the corresponding monoethyl ester per 100 parts by weight is chosen

25 for the embodiment example. A metal complex catalyst, for example zinc octoate, is added to the casting resin system as an accelerator. Other additives customary for casting resins can also be admixed in small amounts. Other possible accelerators are, for example, colourless

30 metal chelate compounds, preferably of Al, Zn or Zr with anions of organic acids, for example with acetate, acrylate, benzoate, salicylate, octoate or acetylacetonate.

35 The curing is carried out at 150°C, for example, and has ended completely after four hours.

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The relatively high heat resistance of these moulded materials is confirmed by TT/DTA experiments up to 800°C. A two-stage degradation mechanism is observed in these. With increasing siloxane content, the rate of degradation decreases and the particular pyrolysis and the combustion residue increases.

The moulded materials are clear and transparent and all pass the soldering bath test (3 x heating to 260°C for 5 seconds each time).

Glass transition temperatures T_g and moduli of elasticity are determined with the aid of solid cured components of moulded materials:

Influence of the siloxane content on the T_g and modulus of elasticity:

	V0	V1	V2	V3
Condensate content (%)	0	20	50	80
Modulus of elasticity (20°C) (N/mm ²) [sic]	2,800	2,750	2,500	2,200
T_g ($\tan\delta_{\max}$) (°C)	177	170	153	99
$\tan\delta(T_g)$	0.976	0.855	0.654	0.601

The absorption of water (7 days at 23°C) is reduced for all the moulded materials produced according to the invention compared with V0.

A multichip array covered with the casting resin according to the invention is tested at operating temperatures of 125°C, and inbetween is subjected to 100 temperature cycles of -55 to +125°C and finally twice to soldering conditions of heating to 260°C for 5 seconds each time. No cracks or other changes at all occur in the covering. The component is unimpaired in its electrical and optical properties.

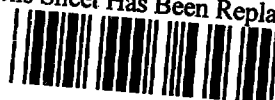
The positive properties of the casting resin system according to the invention are achieved here not only with the embodiment example given by way of example



but also with any desired siloxane condensate and with any desired epoxides. Suitable casting resin system [sic] can be mixed together with aliphatic and aromatic glycidyl ethers, in particular those based on bisphenol
5 A and F, and with corresponding glycidyl esters, aliphatic and cycloaliphatic epoxides or any other desired epoxides, for example those obtained by epoxidation of unsaturated compounds.

Moulded materials dyed with appropriate dyestuffs
10 also still show no change in colour at all after months.

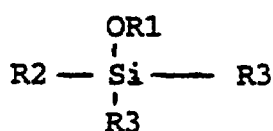
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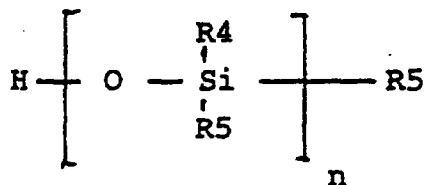


Patent claims

1. Siloxane-containing casting resin system which comprises a resin component A and at least one carboxylic acid anhydride as the curing component B, the A-component of which comprises
 - 5 to 95 % by weight of epoxyalkylsiloxane and
 - 95 to 5 % by weight of epoxide.
2. Casting resin system according to Claim 1, in which the B-component comprises anhydrides of aromatic and cycloaliphatic di- and tetracarboxylic acids.
3. Casting resin system according to Claim 1 or 2, in which the B-component comprises, for flexibilization, a carboxylic acid anhydride which is at least partly esterified with alcohols.
4. Casting resin system according to one of the preceding claims, in which the epoxyalkylsiloxane is the reaction product of a silanol with an epoxyalkoxysilane.
5. Casting resin system according to Claim 4, in which the epoxyalkylsiloxane is obtained by reaction of the epoxyalkoxysilane (1) with the silanol (2)



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wherein

- R1 is an alkyl radical having 1 to 6 C atoms or an aryl radical,
- R2 is a glycidylalkyl, epoxyalkyl or an epoxycycloalkyl radical, the two radicals
- R3 independently of one another are OR1 or R2 or another alkyl or aryl radical,
- R4 is alkyl or aryl, the radicals
- R5 independently of one another are OH or R4 and
- n is an integer, where $1 \leq n \leq 12$.

6. Process for the preparation of a siloxane-containing casting resin system comprising the following





steps:

- a) reaction of the epoxyalkoxysilane 1 with the silanol 2, an epoxysiloxane being obtained,
 - b) mixing of the epoxysiloxane with a cycloaliphatic or aromatic epoxide in a weight ratio of 1 : 20 to 20 : 1,
 - c) provision of a B-component comprising a carboxylic acid anhydride.
7. Process according to Claim 6, in which step a) is carried out in the presence of a neutral condensation catalyst in the range of pH 5 to pH 8.
8. Process according to Claim 6 or 7, in which, after step b), the casting resin system is subjected to a heat treatment in vacuo.
9. Process according to one of Claims 6 to 8, in which the reaction according to step a) is carried out under stoichiometric conditions, so that one alkoxy group in 1 is available per OH group in 2 capable of condensation.
10. Use of the casting resin system according to one of the preceding claims for covering optoelectronic components.

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Abstract

Siloxane-containing casting resin system.

A casting resin system, the resin component A of which comprises a mixture of epoxyalkylsiloxanes and cycloaliphatic epoxide, is proposed for covering optoelectronic component [sic]. The casting resin system can be cured with a carboxylic acid anhydride as the curing component B to give transparent and colour-stable moulded materials.

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Temperature- and colour-stable casting resins are required as a covering for optoelectronic components capable of SMT, for example for optoelectronic multichip arrays.

Transparent colour-stable casting resins are known, for example, from DE 26 42 465. They are preferably employed for sheathing LEDs and LEDs capable of SMT, so-called TOPLEDs. These resins are of only limited suitability for covering components of large surface area, such as, for example, for covering the abovementioned multichip arrays. Coverings of large surface area of the known resins show cracks and adhesion problems which occur, in particular, after temperature shock treatment or after the solder heat required for soldering. These can impair the optical and/or electrical properties of the optoelectronic components.

The object of the present invention is therefore to provide a casting resin which is suitable for covering optoelectronic components, with which a heat-stable covering which is also colour-stable over a relatively long life can be produced, and which avoids the disadvantages of the casting resins known for optoelectronic components. In particular, the moulded material should resist high continuous operating temperatures of 125°C and the exposure to heat during the soldering process.

This object is achieved according to the invention by a casting resin system according to Claim 1. Further embodiments of the invention, in particular a process for the preparation of the casting resin system and a preferred use, are to be found in the sub-claims.

Surprisingly, it has been found that the epoxy resin systems according to the invention modified with siloxane solve the abovementioned problem in an outstanding manner. The casting resins, or their A-components,

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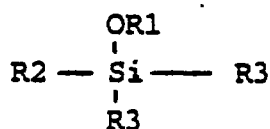
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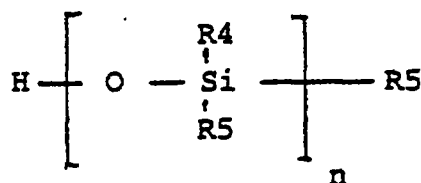
are water-clear and transparent. They can be stored at room temperature for several months without a substantial increase in their viscosity or without a reduction in the epoxide content, and can be processed as conventional epoxy casting resins. The chemistry and the reactivity of the epoxy resin is not adversely influenced by by [sic] the epoxyalkoxysiloxanes.

Although the moulded materials which can be obtained from the casting resin system according to the invention by curing are flexibilized due to the addition of the epoxyalkoxysiloxane, compared with the siloxane-free moulded materials, they do not have the disadvantages usually associated with this property. Thus, the glass transition temperature of the moulded materials is only insignificantly lower than that of a siloxane-free comparison resin. The modulus of elasticity also remains high, while the elastic properties are improved. The heat resistance, which manifests itself in particular in a lower rate of thermal degradation and in a higher decomposition temperature, is improved. The moulded materials here are clear and transparent. In the accelerated ageing process, they display a high colour stability and meet the required specifications.

A particularly advantageous casting resin system is obtained if the epoxysiloxane contained in the A-component to the extent of 5 to 95 % by weight is prepared by condensation of an epoxyalkoxysilane 1 with a silanol 2



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This reaction can be carried out in a simple one-pot reaction under normal conditions with respect to
10 atmosphere and pressure and with a minimum expenditure on the process to give reproducible siloxane condensation products in a very short time. The primary products are readily compatible and can easily be mixed in all proportions with commercially obtainable epoxides. Such
15 mixtures are stable to storage and can be crosslinked with the carboxylic anhydrides customary for casting resin technology.

The epoxyalkoxysilane 1 carries 1 to 3 alkoxy groups which are capable of condensation. The radical R1
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Monomeric epoxyalkoxysilanes 1 are preferred, because of their easier availability, but the reaction is
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Longer-chain silanols 2 which contain the OH groups in the alpha-position, alpha- and omega-position or in the chain are relatively easily available and also
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radicals, however, an increasing incompatibility of the condensation products (epoxyalkylsiloxanes) may result with increasing chain length, which may render their use in the casting resin system according to the invention more difficult or impossible.

The radical R2 containing epoxide groups in the starting compound 1 is bonded to silicon via a C atom and can otherwise be chosen freely. R2 can be a glycidylalkyl or an epoxyalkyl epoxyaryl or epoxycycloalkyl group, depending on the availability of the corresponding epoxyalkoxysilane. The corresponding glycidyloxy compounds which are obtained by reaction of correspondingly reactive compounds with epichlorohydrin are readily available.

A condensation catalyst may be necessary to assist the reaction, depending on the reactivity of the starting substances, which may be hindered electronically and sterically. There are no limitations on the catalyst in respect of the reaction itself, so that any desired condensation catalyst is suitable. A prerequisite is, however, that the catalyst does not influence the epoxide group and likewise does not impair the storage properties and the curing process of the casting resin system. An ideal catalyst therefore has neither a decidedly basic nor a decidedly acid reaction, and in the ideal case is neutral.

The reaction of the starting substances 1 and 2 can be carried out in solution or as a bulk reaction.

Preferred reaction temperatures are between 80 and 150°C, volatile reaction products preferably being blown out in an open reaction vessel by passing in a stream of inert gas. The storage life and stability both of the epoxyalkylsiloxane and of the casting resin system are increased in this way. The storage stability of the casting resin system can be increased by heating the A-component under an applied vacuum.

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The process for the preparation of the casting resin system according to the invention is described in more detail below with the aid of embodiment examples. Preparation of the siloxane-modified A-component:

3-Glycidylpropyltrimethoxysilane (GPT), which is already known as an adhesion promoter for epoxy resins and is admixed to customary epoxy resins in amounts of about 0.25 per cent, is used as the alkoxy silane 1. α,ω -Propylphenylsilanediol (PPS) is chosen as the silanol 2.

57.8 g of PPS are added in portions to 11.8 g of GPT (50 mmol) at 120°C in the course of 15 minutes. The amount of silanol is chosen such that amounts of OH groups equivalent to the methoxy functions are available for the condensation. The solid PPS dissolves, with evolution of methanol, and a highly viscous siloxane condensate forms. During this procedure, the reaction mixture is stirred at 120°C for a total of 4 hours, while distilling off methanol and passing over nitrogen at the same time. This methanol-containing condensate comprises various siloxane products and is processed directly with the desired amount of epoxy casting resin at 120°C to give a storage-stable siloxane-containing A-component.

For this, an epoxyalkylsiloxane (siloxane condensate) prepared as above is mixed in various mixing ratios with the cycloaliphatic epoxide CY 179 (3,4-epoxycyclohexylmethyl 3',4'-epoxycyclohexanecarboxylate, Ciba-Geigy).

For example, the viscosity at 60°C and the epoxide value of an A-component comprising four parts of siloxane condensate and one part of CY 179 change only slightly after storage at 60°C for 6 months. During this period, the viscosity rises from 120 to 160 mPas and the epoxide value drops from 0.581 to 0.574 mol/100 g.

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Production of a transparent moulded material

15 The content of cycloaliphatic epoxide determines the viscosity and the epoxide value and thus the process-ability of the A-component prepared by mixing, to which a stoichiometric amount of B-component is added, according to its epoxide content. This comprises a carboxylic acid anhydride, which can be partly esterified with an

20 alkyl alcohol. Suitable anhydrides are, for example, (methyl-)tetrahydrophthalic anhydride or (methyl-)hexahydrophthalic anhydride. Hexahydrophthalic anhydride which is mixed with 13 parts by weight of the corresponding monoethyl ester per 100 parts by weight is chosen

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Influence of the siloxane content on the T_g and modulus of elasticity:

	V0	V1	V2	V3
Condensate content (%)	0	20	50	80
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$\tan\delta(T_g)$	0.976	0.855	0.654	0.601

The absorption of water (7 days at 23°C) is reduced for all the moulded materials produced according to the invention compared with V0.

A multichip array covered with the casting resin according to the invention is tested at operating temperatures of 125°C, and inbetween is subjected to 100 temperature cycles of -55 to +125°C and finally twice to soldering conditions of heating to 260°C for 5 seconds each time. No cracks or other changes at all occur in the covering. The component is unimpaired in its electrical and optical properties.

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Moulded materials dyed with appropriate dyestuffs also still show no change in colour at all after months.

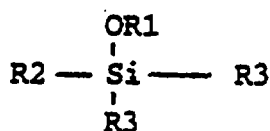
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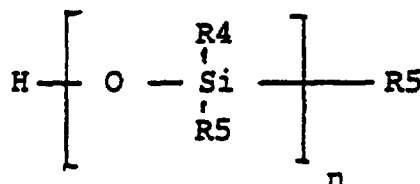


Patent claims

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- 5 to 95 % by weight of epoxyalkylsiloxane and 95 to 5 % by weight of epoxide.
2. Casting resin system according to Claim 1, in which the B-component comprises anhydrides of aromatic and cycloaliphatic di- and tetracarboxylic acids.
3. Casting resin system according to Claim 1 or 2, in which the B-component comprises, for flexibilization, a carboxylic acid anhydride which is at least partly esterified with alcohols.
- 15 4. Casting resin system according to one of the preceding claims, in which the epoxyalkylsiloxane is the reaction product of a silanol with an epoxyalkoxysilane.
5. Casting resin system according to Claim 4, in which the epoxyalkylsiloxane is obtained by reaction of
- 20 the epoxyalkoxysilane (1) with the silanol (2)



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wherein

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6. Process for the preparation of a siloxane-containing casting resin system comprising the following

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steps:

- a) reaction of the epoxyalkoxysilane 1 with the silanol 2, an epoxysiloxane being obtained,
 - b) mixing of the epoxysiloxane with a cycloaliphatic or aromatic epoxide in a weight ratio of 1 : 20 to 20 : 1,
 - c) provision of a B-component comprising a carboxylic acid anhydride.
7. Process according to Claim 6, in which step a) is carried out in the presence of a neutral condensation catalyst in the range of pH 5 to pH 8.
8. Process according to Claim 6 or 7, in which, after step b), the casting resin system is subjected to a heat treatment in vacuo.
9. Process according to one of Claims 6 to 8, in which the reaction according to step a) is carried out under stoichiometric conditions, so that one alkoxy group in 1 is available per OH group in 2 capable of condensation.
10. Use of the casting resin system according to one of the preceding claims for covering optoelectronic components.

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Date: 03 JUL 1996

Abstract

Siloxane-containing casting resin system.

A casting resin system, the resin component A of which comprises a mixture of epoxyalkylsiloxanes and cycloaliphatic epoxide, is proposed for covering optoelectronic component [sic]. The casting resin system can be cured with a carboxylic acid anhydride as the curing component B to give transparent and colour-stable moulded materials.

This is the replacement sheet

Date: 03 JUL 1996