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(54) Title: HIGH SPEED DVDS

(57) Abstract: This invention relates to DVDs made from polycarbonate and a special UV curable bonder material consisting of A) an urethane acrylate oligomer as binder, which is the reaction product of i) one or more polyisocyanates and ii) one or more hydroxyfunctional lactone ester (meth)acrylates having a number average molecular weight of from 200 g/mol to 2000 g/mol and having the formula: $\text{CH}_2 = \text{C}(\text{R}^1) - \text{C}(\text{O}) - \text{O} - \text{R}^2 - [\text{O} - \text{C}(\text{O}) - \text{R}^3]_n - \text{OH}$ wherein, n is an integer from 1 to 5, R^1 is hydrogen or methyl, R^2 represents an alkylene group or substituted alkylene group having from 2 to 10 carbon atoms and R^3 represents a straight or branched chain alkylene group of from 3 to 8 carbon atoms, and which may be substituted with one or more alkyl groups having from 1 to 12 carbon atoms, B) a reactive thinner, comprising of mono functional acrylates and/or (meth)acrylates C) a photoinitiator and D) optional additives which offers improved performance at high data transfer rates.

WO 2011/092200 A1

High Speed DVDs

The present invention describes the usage of special UV bonders for the production of DVDs (digital versatile discs), which facilitate improved focusing and tracking performance at high data transfer rates.

- 5 The development of optical data storage is mainly characterized by the increase of the storage capacity per disc from one generation to the next generation. This can be exemplified e.g. by the 0.7 GByte capacity per disc in the Compact Disc (CD) format, the 4.7 GByte capacity of a single layer Digital Versatile Disc (DVD) and the 25 GByte capacity of a single layer Blu-ray Disc (BD). As can be seen in the case of DVD and BD those capacities can be increased by introducing e.g. double layer discs or discs with even more layers or double side accessible discs.
- 10

- An additional important point is the data transfer rate during readout and/or recording of a disc. Due to the fact that the data are readout and/or recorded sequentially, the increase of the storage density lead to increasing readout and/or recording times of a full disc, despite the fact that the pits or recorded marks become smaller from one generation to the next generation. This lack of transfer rate is compensated for that the reference speed for 1X recording and readout for each format increases from one generation to the next generation. To increase the data transfer rate within a format the increase of the spinning speed of the disc facilitates high speed recording and readout.
- 15

- For the CD-format drives and discs were developed which can reach 52X of data transfer rates in comparison to the 1X reference speed. For 52X speed the spinning speed is about 10500 rounds per minute (RPM) as for a 0.7 GByte capacity disc the linear reference speed is 1.2 m/s.
- 20

- For the DVD-format drives and discs were developed which can access up to 22X of data transfer rate in comparison to the 1X reference speed. For 22X speed the spinning speed is about 12830 RPM. High rotation speeds will reduce recording times dramatically as can be seen from the following table 1.
- 25

Table 1: Writing speeds for DVD with 1X, that is 1350 kBytes/s, in the first drives and media models. More recent models work at 18X or 20X that means at 18 or 20 times higher maximum data transfer rate. Note that for CD drives, 1X means 150 kBytes/s, which is approximately 9 times slower than 1X DVD.

DVD drive speeds				
Drive speed	Max. data transfer rate		~Write time (min)	
	(Mbit/s)	(MB/s)	Single Layer	Double Layer
1X	10.55	1.35	61	107
2X	21.09	2.70	30	54
2.4X	27.43	3.51	24	42
4X	42.19	5.40	15	27
6X	63.30	8.10	11	18
8X	84.38	10.80	8	14
12X	126.60	16.20	6	11
16X	168.75	21.60	4	7
18X	189.90	24.30	3	5
20X	211.00	27.00	3	4

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For the BD-format discs and drives exist that support up to 8X of data transfer rate in comparison to the 1X reference speed. For 8X speed the spinning speed is about 6480 RPM as the linear velocity for the 1X reference speed is 4.92 m/s for a 25 GByte/layer capacity disc. In the laboratory 12X recording speed was already demonstrated, which corresponds to 9720 RPM spinning speed of the disc (In proceedings of the ISOM 2006 (International Symposium of Optical Memory) held in Takamatsu, Japan, "Up to 12X BD-R (Cu:Si) Recording", R.G.J.A. Heslen, J.H.G. Jaegers, A.P.G.E. Janssen, J. Rijpers, P.R.V. Sonnevile, J.J.H.B. Schleipen, Philips Research Laboratories, High Tech Campus 34, 5656 AE Eindhoven, The Netherlands).

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At such high rotation speeds tracking and focusing of the laser becomes crucial to record and read data in high quality. In such cases it can happen that the disc reader or writer reduces the disc rotation speed to gain better data signal quality at lower error rates. Thus high rotation speeds cannot be achieved.

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E.g. DVD writer producer often check the quality of the tracking error (TE) and focusing error (FE) signals at high speeds (e.g. 16X) before doing power calibration for recording. If TE or FE are above the writer specific limits recording will be done at a much lower speed. Examples for freeware tools to check such TE and FE quality are Plectools for Plector writers and Qscan that works for most BenQ and Philips writers.

Proper tracking and focusing is strongly related to the evenness of the disc surface as for the optical pickup system the working distance and the alignment of its optical axis to the data tracks changes with time if the disc rotates underneath. That means that low tangential deviation of the disc is important, which means a periodic and synchronized change of the above mentioned properties for the pick up head. High requirements on low tangential and radial deviation narrow the processing window for high speed capable DVD production and means extra burden for the disc producer.

But also modal oscillations can gain significant amplitudes with respect to the focal length at high rotation speeds. Such modal oscillations also generate non synchronized transient changes of working distance and alignment which will make tracking and focusing difficult at high rotation speeds, as the tracking and focusing servos are bandwidth limited and can not follow large and highly frequent distortions fast enough. The modal oscillation is characterized by its modal frequency f_n which is related to the geometry of the disc and the ration of Youngs modulus E and mass density ρ according to f_n proportional to $(E/\rho)^{0.5}$ (see also (3)). The quality factor Q (see formula (4)) is related to $\tan\delta$ via $Q=3/\tan\delta$. In that sense Q can be used as a measure of the damping like $\tan\delta$. Low Q means high damping as $\tan\delta$ is high. In general E and Q show a distinct dependency on frequency f . The group of lowest frequency modal oscillations of a 12 cm diameter and 1.2 mm thick disc made from polycarbonate lay in the range of 130 Hz to 170 Hz [S. Subramanian, R. Hariharan, B. Landa, T. P. Feist, in Optical Data Storage 2000, Proceedings of SPIE Vol. 4090 (2000), pp 323 ff, WO 0048172A2 Comparative example 1]. The next group modal oscillations occur around 280 Hz. As the spinning speed at 16X is about 155 Hz such modal oscillations can be easily excited during disc operation.

WO 0048172A2 discloses solutions to suppress the influence of modal oscillations on high density data storage ($> 5 \text{ Gbit/in}^2$ areal density $> \text{DVD density}$) media by controlling stiffness and damping. However due to the fact that the information layer is at a first surface of that media the stiffness and damping requirements could be fulfilled by nontransparent substrate materials. The disclosed solutions employ sandwich injection molding processes that can not be used in economic DVD production.

EP 1 158 024 A1 describes a vibration-damping thermoplastic resin composition comprising a) 50 – 90 wt.-% of an amorphous thermoplastic resin having a loss $\tan\delta$ of 0,01 to 0,04 and a deflection temperature under load of not lower than 120°C and b) 50 to 10 wt.% of a methyl methacrylate resin wherein the article molded therefrom has certain physical properties. The disclosed solution achieves an increased damping and uses however polymer blends of special polycarbonates with methyl methacrylate. Optical clarity of the disclosed solutions will not be good enough for DVD applications.

WO 2006046864A1 discloses the use of urethane acrylates obtained from diols, diisocyanates and hydroxylgroup containing (meth)acrylates in combination with epoxydiacrylate, monofunctional and difunctional acrylic acid esters as adhesive for optical discs. However difunctional acrylic acid esters as well as epoxyacrylates proved to be not suitable in bonders for DVD discs at high spinning speeds (see comparative example 16).

CN 1456621A discloses a UV-curable coating suitable for DVDs. These UV-curable coatings comprising an aminoformate-acrylate ester as active oligomer, mono- and difunctional (meth)acrylates, a photoinitiator and optional additives.

There still exists the necessity for DVD discs that can provide at high spinning speeds improved tracking and focusing operation. The usage of standard DVD bonder material already impose tight production margins to keep the TE and FE signals within acceptable limits at high recording speeds. Often discs are not accepted by the DVD writers for 16X or even higher X recording because of poor tracking or focusing behavior.

It was therefore the task to provide DVDs that show wider margin with respect to tracking and focusing at high rotation speeds.

Surprisingly it was found that the usage of a special UV curable bonder can solve the above described problem. The present invention is directed to DVDs made from polycarbonate, with a special bonder material and optionally additional layers.

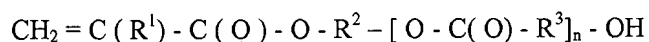
The UV curable bonder material is in general a spin coatable formulation consisting of

A) an urethane acrylate oligomer as binder, which is the reaction product of

i) one or more polyisocyanates and

ii) one or more hydroxyfunctional lactone ester acrylates having a number average molecular weight of from 200 g/mol to 2000 g/mol and having the formula:

- 5 -



wherein n is an integer from 1 to 5, R¹ is hydrogen or methyl, R² represents an alkylene group or substituted alkylene group having from 2 to 10 carbon atoms and R³ represents a straight or branched chain alkylene group of from 3 to 8 carbon atoms, and
 5 which may be substituted with one or more alkyl groups having from 1 to 12 carbon atoms.

B) a reactive thinner, comprising monofunctional (meth)acrylates,

C) a photoinitiator and

D) optional additives.

10 Additionally it was found that such special UV curable bonder when used in DVD production results in discs with a Q factor of less than 130 measured at a frequency of 270 Hz.

In a short term DVD discs made with bonders that enable an improved damping at frequency levels of around 200 Hz to 300 Hz at room temperature, provide significantly improved tracking and focusing behavior at recording speed of 16X or above.

15 In the context of this specification, "(meth)acrylate" relates to corresponding acrylate or (meth)acrylate functions or to a mixture of the two.

The urethane acrylate oligomer A) may be produced according to the methods described in EP-A 1 650 241 or EP-A 1 671 995.

The polyisocyanate component (i) in the urethane acrylate oligomer A) comprises

- 20
- polyisocyanates selected from the group comprising aromatic, araliphatic, aliphatic or cycloaliphatic polyisocyanates or mixtures of such polyisocyanates, for example 1,3-cyclohexane diisocyanate, 1-methyl-2,4-diisocyanato-cyclohexane, 1-methyl-2,6-diisocyanato-cyclohexane, tetramethylene diisocyanate, 4,4'-diisocyanatodiphenylmethane, 2,4'-diisocyanatodiphenylmethane, 2,4-diisocyanatotoluene, 2,6-diisocyanatotoluene,

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 - α,α,α',α'-tetra-methyl-m- or -p-xylylene diisocyanate, 1,6-hexamethylene diisocyanate, 1-isocyanato-3,3,5-trimethyl-5-isocyanatomethylcyclohexane (isophorone diisocyanate or IPDI), 4,4'-diisocyanato-dicyclohexylmethane, 4-isocyanatomethyl-1,8-octane diisocyanate (triisocyanatononane, TIN) (EP-A 928 799) and mixtures thereof. Also suitable

are homologues or oligomers of the mentioned polyisocyanates with biuret, carbodiimide, isocyanurate, allophanate, iminooxadiazinedione and/or uretdione groups, mixtures thereof with one another and mixtures with the polyisocyanates mentioned above. Preference is given to 1,6-hexamethylene diisocyanate, 1-isocyanato-3,3,5-trimethyl-5-isocyanatomethylcyclohexane (isophorone diisocyanate or IPDI) and 4,4'-diisocyanato-dicyclohexylmethane and mixtures thereof with one another. Preference is given also to homologues or oligomers of 1-isocyanato-3,3,5-trimethyl-5-isocyanatomethylcyclohexane (isophorone diisocyanate or IPDI) and 4,4'-diisocyanato-dicyclohexylmethane with biuret, carbodiimide, isocyanurate, allophanate, iminooxadiazinedione and/or uretdione groups, mixtures thereof with one another and mixtures with the preferred polyisocyanates mentioned above.

- Prepolymers of hexamethylene diisocyanate, made from polyesterdiols and/ or polyetherdiols and hexamethylene diisocyanate
- Prepolymers of dicyclohexylmethane diisocyanates, made from polyesterdiols and/ or polyetherdiols and dicyclohexylmethane diisocyanate

Particularly preferred as polyisocyanate component i) are oligomers of hexamethylenediisocyanate and/or dicyclohexylmethanediisocyanate.

Most preferred as the hydroxyfunctional lactone ester acrylate ii) in the urethane acrylate oligomer A) is the reaction product from hydroxyethylacrylate, hydroxypropylacrylate or hydroxybutylacrylate with ϵ -Caprolactone.

The reactive thinner B) comprises monofunctional (meth)acrylates.

Preferred compounds B) are selected from the group comprising alkoxyated alkyl (meth)acrylates, monofunctional esters of (meth)acrylic acid and C1 to C10- alkoxyated phenols, alkyl (meth)acrylates, cycloaliphatic (meth)acrylates and heterocyclic (meth)acrylates.

Particularly preferred compounds B) comprises compounds selected from the group of C1- to C4- alkoxyated C1- to C4- alkyl (meth)acrylates, monofunctional esters of (meth)acrylic acid and C1- to C4- alkoxyated phenols, C1- to C10- alkyl (meth)acrylates, C4- to C10- cycloaliphatic (meth)acrylates and C4- to C6 heterocyclic (meth)acrylates.

- 7 -

The most particularly preferred compounds B) comprises compounds selected from the group of phenoxy-ethylacrylate, phenole-(2,5-diethoxy) acrylat, 2-(2-ethoxyethoxy)ethylacrylate, isobornyl acrylate and tetrahydrofurfuryl acrylate.

Typical photoinitiators C) comprising hydroxyphenyl ketones e.g. (1-hydroxycyclohexyl)phenyl ketone (e.g. Irgacure[®] 184, Ciba, Lampertheim, DE), 1-hydroxydimethylphenyl ketone (e.g. Darocur[®] 1173, Ciba, Lampertheim, DE), acylphosphinoxides, e. g. 2,4,6-trimethyl-benzoyl-diphenylphosphine oxide (e.g. Darocur[®] TPO, Ciba, Lampertheim, DE), phenylbis-(2,4,6-trimethylbenzoyl)phosphine oxide (e.g. Irgacure[®] 819, Ciba, Lampertheim, DE), phenylglyoxylates, e. g. phenylglyoxylic acid (methyl)ester (e.g. Irgacure[®] MBF, Ciba, Lampertheim, DE) and benzyldimethylketales (e.g. Irgacure[®] 651, Ciba, Lampertheim, DE).

Additives, binders and auxiliary substances D) known in coating and adhesive technology can be added to or combined with the bonder material. Such substances are flow and wetting additives, slip additives, fillers, nanoparticles, light-stabilising particles, anti-yellowing additives, thickeners, and additives for reducing the surface tension.

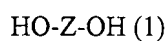
According to the invention, the UV curing bonder materials are formulated to such a ratio between components A) to B) that the viscosity at 25 °C is about 200 to 1000 mPas, preferably about 350 to 800 mPas and particularly preferred about 450 to 650 mPas.

The photoinitiator C) in the UV curing bonder composition according to the invention is contained in amounts of 1 to 10 wt.-%, preferably 2 to 8 wt.-%, particularly preferably 3 to 6 wt.-% based on all polymerizable compounds.

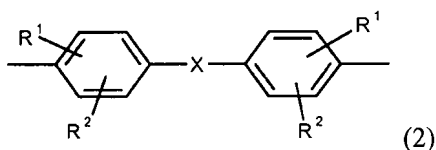
General description of the substrate material

The polycarbonate resins are generally obtained by solution polymerization or melt polymerization of an aromatic dihydroxy compound and a carbonate precursor. Any aromatic dihydroxy compound is acceptable if discs made out of the respective polycarbonate satisfy the requirements of the high rotation speed test wherein said test is described in the experimental part of the specification and / or the respective polycarbonate satisfies the above relative solution viscosity ranges

Preferred aromatic dihydroxy compounds are compounds of formula (1)



in which Z denotes a radical of the formula (2)



in which

R¹ and R² independently of each other represent H or C₁-C₈-alkyl, preferably H or C₁ - C₄-alkyl,
 5 in particular hydrogen or methyl and

X represents a single bond, C₁ - C₆-alkylene, C₂ - C₅-alkylidene or C₅- C₆- cycloalkylidene, which may be substituted by C₁ - C₆-alkyl, preferably methyl or ethyl, with the proviso that R¹ and R² represent hydrogen if X represents 3,3,5- trimethylcyclohexylidene.

Most preferably X represents isopropylidene and R¹ and R² represents hydrogen.

10 Aromatic dihydroxy compounds are generally known or can be prepared according to generally known methods. The interfacial or melt polymerization of polycarbonate is also generally known and described in numerous publications e.g. WO 2004/063249 A1, WO 2001/05866 A1, WO 2000/105867, US-A 5,340,905, US 5,097,002, US-A 5,717,057.

The polycarbonate resin may be a homopolycarbonate obtained by homopolymerizing the above
 15 aromatic dihydroxy compound or a copolycarbonate obtained by copolymerizing two or more aromatic dihydroxy compounds mentioned above. The polycarbonate based on bisphenol A as diphenol monomer may contain up to 20 wt.-%, preferably up to 10 wt.-%, more preferably up to 5 wt.-%, based on the amount of diphenols used for the production of polycarbonate, additional diphenol monomers selected from the group consisting of 1,1-bis(4-hydroxyphenyl)-
 20 3,3,5-trimethylcyclohexan, α,α'-bis(4-hydroxyphenyl)m-diisopropylbenzen or 4,4'-dihydroxy-biphenyl or 2-Phenyl-3,3-bis(4'-hydroxyphenyl)phthalimide.

The reaction by the solution method is usually a reaction between the aromatic dihydroxy compound and phosgene, and is usually carried out in the presence of an acid coupling agent and an organic solvent. As the acid coupling agent, alkali metal hydroxides, such as sodium hydroxide
 25 or potassium hydroxide, or amine compounds, such as pyridine, are used. As the organic solvent, halogenated hydrocarbons, such as methylene chloride or chlorobenzene or mixtures of such hydrocarbons, are used. For accelerating the reaction, a catalyst such as tertiary amines, quaternary ammonium compounds or quaternary phosphonium compounds which are exemplified by triethylamine, N-ethyl-piperidine, tetra-n-butylammonium bromide, or tetra-n-

butylphosphonium bromide respectively may also be used. Preferably, the reaction temperature is routinely 0 to 40° C, with a reaction time being 10 seconds to 5 hours and with a pH during the reaction being not less than 9. The reaction can be a batch process or a continuous process.

In the polymerization reaction, terminal capping agents for adjusting the relative solution viscosity are usually also employed. These terminal capping agents used may be monofunctional phenols. The polycarbonate resin obtained has its terminal capped by a monofunctional phenol based group so that it is superior in thermal stability to polycarbonate resin not obtained as described above. The monofunctional phenols are generally phenols or lower alkyl substituted phenols like phenol, p-tert-butylphenol, p-cumylphenol, isooctylphenol or long-chain alkyl phenols like decyl phenol, dodecyl phenol, hexadecyl phenol, octadecyl phenol, eicosyl phenol, docosyl phenol and triacontyl phenol. Preferred terminal capping agents are phenol, p-tert-butylphenol, p-cumylphenol and 3-n-pentadecylphenol (CAS # 501-24-6).

The terminal capping agent is introduced in an amount to achieve the appropriate relative solution viscosity. The terminal capping agent may be used alone or in combination with other above mentioned phenol compounds.

The reaction by the melting method is usually an ester exchange reaction between an aromatic dihydroxy compound mentioned above and a carbonate ester, and is carried out by a method consisting in mixing the aromatic dihydroxy compound and the carbonate ester in the presence of an inert gas under heating and in distilling off the generated alcohol or phenol. Although the reaction temperature differs with e.g., the boiling point of the generated alcohol or phenol, it is usually 120 to 350° C. During the latter half of the reaction, the reaction system is reduced in pressure to approximately 1.33×10^3 to 13.3 Pa to facilitate distillation of the generated alcohol or phenol. The reaction time is usually 1 to 6 hours.

Among the carbonate esters, there are esters, such as C6 to C10 aryl groups or aralkyl groups or C1-4 alkyl groups, that may occasionally be substituted, specifically, diphenyl carbonate, bis(chlorophenyl) carbonate, dinaphtyl carbonate, bis(diphenyl) carbonate, dimethyl carbonate, diethyl carbonate and dibutyl carbonate. Of these, diphenyl carbonate is most preferred.

For accelerating the polymerization, polymerization catalysts may also be used. As these polymerization catalysts, those catalysts routinely used for esterification or ester exchange reactions, such as alkali metal compounds, e.g., sodium hydroxide, potassium hydroxide, or sodium or potassium salts of dihydric phenols, alkali earth metal compounds, such as calcium hydroxide, barium hydroxide or magnesium hydroxide, nitrogen-containing basic compounds, such as tetramethyl ammonium hydroxide, tetraethyl ammonium hydroxide, trimethylamine or triethylamine, alkoxides of alkali metals or alkali earth metals, phosphorous-containing basic com-

pounds like tetraphenylphosphoniumphenolat or organic acid salts of alkali metals or alkali earth metals, zinc compounds, boron compounds, aluminum compounds, silicon compounds, germanium compounds, organotin compounds, lead compounds, osmium compounds, antimony compounds, manganese compounds, titanium compounds or zirconium compounds, may be used. These catalysts may be used alone or in combination. These catalysts are used in an amount preferably of 1×10^{-8} to 1×10^{-3} equivalent, more preferably 1×10^{-7} to 5×10^{-4} equivalent, to one mol of the dihydric phenol as the starting material.

The aromatic polycarbonate resins may contain tri-or higher functional aromatic compounds or contain branched components in the polymer as a result of the isomerization reaction in polymerization. Examples of the tri-or higher functional aromatic compounds preferably include phlorogrucin, phlorogrucide, tris phenols, such as 4,6-dimethyl-2, 4,6-tris(4-hydroxyphenyl) heptene-2,2,4,6-trimethyl-2,4,6-tris(4-hydroxyphenyl) heptane, 1,3,5-tris(4-hydroxyphenyl) benzene, 1,1,1-tris(4-hydroxyphenyl) ethane, 1,1,1-tris(3,5-dimethyl-4-hydroxyphenyl) ethane, 2, 6-bis(2-hydroxy-5-methylbenzyl)-4-methyl phenol, and 4-(4-[1,1-bis(4-hydroxyphenyl) ethyl] benzene)- α,α -dimethylbenzyl phenol, tetra(4-hydroxyphenyl) methane, bis(2,4-dihydroxyphenyl) ketone, 1,4-bis(4,4-dihydroxytriphenyl methyl) benzene, trimellitic acid, pyromellitic acid, benzophenone tetracarboxylic acid and acid chlorites thereof. In case tri- or higher functional aromatic compounds are used, 1,1,1-tris(4-hydroxyphenyl) ethane and 1,1,1-tris(3,5-dimethyl-4-hydroxyphenyl) ethane, are preferred.

The polycarbonate or the copolycarbonate are characterized by the relative solution viscosity, determined in a polymer solution in dichloromethane, measured with an Ubbelohde capillary viscosimeter, capillary type 0C. The polymer concentration is 5 g/l, the measurement is done at a temperature of 25 °C. The preferred relative solution viscosity should be in the range of 1.190 to 1.230.

As release agent higher fatty acid ester of a monohydric alcohol or polyhydric alcohol may be added to the thermoplastic resin of the present invention. By blending a higher fatty acid ester of a monohydric or polyhydric alcohol, releasability from a mold at the time of molding the thermoplastic resin is improved, and a release load in the molding of a disk substrate becomes small, thereby making it possible to prevent the deformation of the disk substrate and pit dislocation caused by a release failure. The melt fluidity of the thermoplastic resin is also improved.

The positive amount of release agents is preferably less than 2000 ppm by weight of the resin, more preferably less than 1000 ppm by weight of the resin, especially preferred less than 500 ppm.

Preferred higher fatty acid esters are glycerol monostearate and glycerol monopalmitate as well as mixtures of them.

Phosphorus-based thermal stabilizers, may be added to the thermoplastic resin also. Suitable phosphorus-based thermal stabilizers are for example, phosphorous acid, phosphoric acid, phosphonous acid, phosphonic acid and esters thereof. Specifically, phosphite compounds, such as triphenyl phosphite, tris nonylphenyl phosphite, tris(2,4-di-tert-butylphenyl) phosphite, tridecyl phosphite, trioctyl phosphite, trioctadecyl phosphite, didecyl monophenyl phosphite, dioctyl monophenyl phosphite, diisopropyl monophenyl phosphite, monobutyl diphenyl phosphite, monodecyl diphenyl phosphite, mono-octyl diphenyl phosphite, bis(2,6-di-tert-butyl-4-methylphenyl) pentaerythritol diphosphite, 2,2-methylenebis(4,6-di-tert-butylphenyl) octyl phosphite, bis (nonylphenyl) pentaerythritol diphosphite and bis(2,4-di-tert-butylphenyl) pentaerythritol diphosphite, and phosphate compounds, such as tributyl phosphate, trimethyl phosphate, tricresyl phosphate, triphenyl phosphate, trichlorophenyl phosphate, triethyl phosphate, diphenyl cresyl phosphate, diphenyl monoorthoxenyl phosphate, tributoxy ethyl phosphate, dibutyl phosphate and diisopropyl phosphate, may be indicated. Additional phosphorus-based thermal stabilizers are for example tetrakis(2,4-di-tert-butylphenyl)-4,4'-biphenylene diphosphonite, tetrakis(2,4-di-tert-butylphenyl)-3,3'-biphenylene diphosphonite and bis(2,4-di-tert-butylphenyl)-4-biphenylene phosphonite. Of these, tris nonylphenylphosphonite, distearyl pentaerythritol diphosphite, bis(2,4-di-tert-butylphenyl) pentaerythritol diphosphite, tris(2,4-di-tert-butylphenyl) phosphite, triphenyl phosphate, trimethyl phosphate, tetrakis(2,4-di-tert-butylphenyl)-4,4'-biphenylene diphosphonite and bis(2,4-di-tert-butylphenyl)-4-biphenylene phosphonite, are preferred. These thermal stabilizers may be used alone or as a mixture.

The amount of these thermal stabilizers, if added, is preferably 0.0001 to 0.05 weight part, more preferably 0.0005 to 0.02 weight part and most preferably 0.001 to 0.01 weight part to 100 parts by weight of the thermoplastic resin composition.

A generally known antioxidant may be added to the thermoplastic resin of the present invention to prevent oxidation. An example of the antioxidant is a phenol-based antioxidant. The amount of the antioxidant is preferably 0.0001 to 0.05 wt.-% based on the thermoplastic resin.

Above mentioned additives as well as other additives like optical stabilizers, colorants, antistatic agents and lubricants may be added to the resin for an optical disk substrate of the present invention in such limits that discs made out of the respective polycarbonate resin composition satisfy the requirements of the high rotation speed test.

Blends of polycarbonate with other thermoplastic resin may also be used as long as the discs made out of the respective blend satisfies the requirements of the high rotation speed test. Other

thermoplastic resin may be selected from the group consisting of acrylic resin, preferably polymethylmethacrylate, polystyrene resins as homopolymer or as co- or blockpolymer with other monomers selected from the group comprising isoprene and butadiene or mixtures thereof, or polycycloolefinic resins preferably polymerization products of olefins, such as moieties with a norbornene structure.

In the preparation of the resin composition of the present invention, it is conceivable that mixing of polycarbonate resins and/or mixing of a polycarbonate resin and other resin is carried out in solution or in the melt. As for means of mixing, in the stage of a polymer solution, a vessel equipped with a stirrer is mainly used, and for the second case equipment like a tumbler, twin-cylinder mixer, Nauter mixer, Banbury mixer, kneading roll or extruder may be used. In either case, any technique may be used and not particularly limited.

Process for the Production of the DVD Substrate

To produce a substrate from the above resin for an optical recording medium, an injection molding machine (including an injection compression molding machine) equipped with a stamper having pits and grooves which satisfy specifications required for an optical recording medium and a surface accuracy is used to form an optical disk substrate by injection molding. The thickness of the disk substrate generally is 0.55 to 0.60 mm. This injection molding machine may be a generally used machine but preferably a machine made from a material whose cylinder and screws have low adhesion to resins, corrosion resistance and abrasion resistance in order to suppress the production of carbide and improve the reliability of a disk substrate. The environment of the molding step is preferably as clean as possible in view of the object of the present invention. It is also important that the material to be molded should be fully dried to remove water and that residence which might cause the decomposition of a molten resin should be avoided.

In the following a production of a standard disc substrate of the optical recording medium of the present invention is described, said method was used in the experimental part and is not limiting to the scope of the invention. A disc substrate with a diameter of 120 mm and a thickness of 0.60 mm is injection molded from polycarbonate using an injection molding machine equipped with a mold and a stamper.

Process for the Production of the Dye and the Reflective Layer

DVD recordable discs use an organic dye as recording layer by means of spin coating process. For this process a fresh dye solution is prepared by solubilizing the dye powder in organic solvents. After the molding process, all substrates are transferred into temperature controlled conveyor to get efficient cooling of both sides of the substrate, ready for further dye coating process.

ess. The inline dye coating units are usually equipped with several automated dye dosing stations for the application of the precise controlled dye layer onto each grooved substrate. The spin coating process shall be strictly under a climate controlled environment. To remove residual solvent from the condensed dye film, the dye coated substrates are fed into the drying oven
5 with infrared lamps.

For the deposition of a highly reflective metal layer on the top of the dye coated substrates, the high rate sputter module is used after drying process. The most common material is silver, or a silver alloy. A powerful and optimized metallizer can offer uniform sputtering throughout the target lifetime. Removal of residual dye on the edge of the dye coated and metallized layer
10 0 substrates by dosing the edge clean solvent after metallizing.

Description of the Bonding Process

Inline process

The metallized and edge-cleaned layer 0 substrate was transferred to the bonding unit, where the bonder material is dosed at a disc by spin coating bonding process. Afterwards another blank
15 layer 1 substrate was placed on top. The bonder was distributed between the two substrates with optimized spin process. The dosage of the bonder is chosen so that the bonder layer thickness achieved by this procedure is between 30 and 60 μm , preferably between 40 and 50 μm .

The discs are exposed to ultraviolet radiation to cure the bonding material by means of a radical polymerization reaction.

EXPERIMENTAL

Testing Procedures

Viscosity of the bonder

The bonder formulation was prepared in a speed mixer (Hauschild DAC 150 FVZ). The viscosity was measured at 25 °C in an Oscillation Rheometer (Firma Anton Paar Physica Modell MCR 301) equipped with an oven CTD 450 which was set to 25 °C. A cone - plate geometry was used. The diameter of the cone was 50 mm and the cone angle was 1°. The viscosity was measured in rotation mode at fixed shear rate of 10 1/s.

Measurement of the Youngs Modulus E and the quality factor Q

To determine the Young's modulus E and the damping related factor Q (quality factor) of the materials in the frequency range from about 10 Hz to 10 KHz, a set up that is analogue to what is described in ASTM E 756-05 (Figure 1) is used. This measurement principle is based on the evaluation of the resonance frequencies f_n of an oscillating beam of the material of interest. To excite the resonance frequencies the beam is clamped on one side to a Piezo-shaker, which is driven by an analog white noise signal with a range stretching from 1 mHz to 10 KHz. The beam response to that excitation e.g. the velocity versus frequency measured with a Laser-Doppler-Vibrometer (LDV) at the tip of the beam that is not clamped to the Piezo-shaker normalized to e.g. the velocity versus frequency of the excitation at the end of the beam that is clamped to the Piezo-shaker is recorded. From this response curve E and Q are calculated as follows:

$$E = \frac{12\rho l^4 f_n^2}{H^2 C_n^2} \quad (3)$$

$$Q = \frac{2f_n}{\Delta f_n} \quad (4)$$

with:

E = Young's modulus of beam material in Pa,

f_n = the resonance frequency for mode n in Hz,

Δf_n = the half-power at full bandwidth (FWHM) of mode n in Hz,

C_n = coefficient for mode n of a clamped-free (uniform) beam,

H = thickness of beam in oscillation direction in meter,

l = length of beam in meter,

5 n = mode number: 1, 2, 3, . . . ,

Q = Quality factor of the beam material, dimensionless,

ρ = mass density of the beam in kg / m^3 .

The beam was cut out of a disc, that was fabricated according to the procedures described above or can be injection molded. The width W of the beam is chosen to be 0.013 m and the length l is chosen, so that the resonance frequencies fit well to the measured frequency range of 10 Hz to 10 kHz. The values of E and Q at 2000 Hz are calculated via linear interpolation using the respective values extracted from formula (3) and formula (4) at the measured f_n below 2000 Hz and the measured f_{n+1} above 2000 Hz.

Mass density

15 The mass density ρ was measured with the Mettler density kit Mettler AT 250 H66765 at room temperature using ethanol as the immersing liquid, on samples cut out of the disks. Or alternatively a rectangular piece of the disc of known width W , known height H and known length l was cut out of the disc. The mass m of the rectangular piece of the disc was determined by weighing with a balance (e.g. Mettler AT 250) and ρ was calculated via $\rho = m / (l \cdot H \cdot W)$.

20 Measurement of the radial and tangential deviation

The radial deviation measures the angular deviation of the disc surface from a reference plane. It is defined as the angle measured in radial direction, between incident light perpendicular to the reference plane and the reflected light. The radial deviation of the disc was measured using an offline optical disk scanner from Basler (Modell: Basler Reference 100). The maximum nominal value measured on the disc is taken.

TE and FE signal evaluation of DVD+R (SL) discs

A DVD writer (Plextor® - PX755A 1.01) was used to evaluate the TE/FE performance. Evaluation was done with the PlexTools Professional V2.32a software that comes with that writer.

Another DVD writer (BenQ® 1650 BCIB) was used to evaluate the TE/FE performance. Evaluation was done with the QScan software that comes with that writer. The measurement has to be done on unrecorded discs at 16X speed. The maximum measured values of TE and FE were taken for each scan of a disc.

- 5 TE/FE test is a write test for blank DVD recordable discs (in simulation mode), measure the mechanical characteristics of the disc. FE (Focus Errors) indicates how well the pickup can focus the laser beam on the disc surface. TE (Tracking Errors) indicates how well the pickup can follow the spiral track of the disc. If the numbers are too high, there is a chance that a write error will happen. In this case the recording should be performed at a lower speed. This test will
10 be performed at the highest recording speed in simulation mode. At the end of the test, the test result will indicate if this disc can be written at the highest speed with the good quality.

Description of the binder A)

Binder AA) is the urethane acrylate Desmolux® XP2513 from Bayer MaterialScience AG, Leverkusen, DE.

- 15 Binder AH) is Ebecryl® 600 from Cytec Surface Specialities SA/NV; Ebecryl® 600 is a standard bisphenol A epoxy acrylate.

Further binders have been synthesized as depicted below. For these syntheses all percentages are by weight unless indicated otherwise.

- 20 The determination of the NCO content in % was undertaken by back titration with 0.1 mol/l hydrochloric acid following reaction with butylamine, on the basis of DIN EN ISO 11909.

The viscosity measurements of the binder were carried out with a plate-plate rotational viscosimeter RotoVisko 1 from Haake, DE in accordance with ISO/DIS 3219:1990. The ambient temperature of 23 °C, prevailing at the time when the experiments were conducted is referred to as RT.

25 Preparation of the urethane acrylate binder AD

- A 2000-ml four-necked glass flask with reflux condenser, heatable oil bath, mechanical stirrer, air traversal (2 l/h), internal thermometer and dropping funnel was charged with 275.1 g of bis-(4-isocyanatocyclohexene) methane (Desmodur® W, Bayer Material Science AG, Leverkusen, DE). 0.50 g of dibutyltin dilaurate (Desmorapid® Z, Bayer MaterialScience AG, Leverkusen,
30 DE) and 1.01 g 2,6- di-*tert*.-butylmethyl phenole were added and the mixture was heated to 60 °C. Subsequently, 722.4 g of Tone M100® (Dow, Schwalbach, DE) were added drop wise at

a rate such that the temperature did not exceed 70 °C. Stirring was continued until the NCO-content dropped below 0.1 %. This gave a colorless resin with a NCO-content of 0.08 % and a viscosity of 15400 mPas (23 °C).

Preparation of the urethane acrylate binder AE

- 5 A 2000-ml four-necked glass flask with reflux condenser, heatable oil bath, mechanical stirrer, air traversal (2 l/h), internal thermometer and dropping funnel was charged with 366.7 g of a HDI biuret (Desmodur[®] N3200, Bayer Material Science AG, Leverkusen, DE). 0.49 g of dibutyltin dilaurate (Desmorapid[®] Z, Bayer MaterialScience AG, Leverkusen, DE) and 1.01 g 2,6-di-*tert.*-butylmethyl phenole were added and the mixture was heated to 60 °C. Subsequently,
- 10 653.6 g of Tone M100[®] (Dow, Schwalbach, DE) were added dropwise at a rate such that the temperature did not exceed 70 °C. Stirring was continued until the NCO-content dropped below 0.15 %. This gave a colorless resin with a NCO-content of 0.12 % and a viscosity of 38500 mPas (23 °C).

Preparation of the urethane acrylate binder AF

- 15 A 2000-ml four-necked glass flask with reflux condenser, heatable oil bath, mechanical stirrer, air traversal (2 l/h), internal thermometer and dropping funnel was charged with 366.7 g of a HDI uretdione (Desmodur[®] N3400, Bayer Material Science AG, Leverkusen, DE). 0.50 g of dibutyltin dilaurate (Desmorapid[®] Z, Bayer MaterialScience AG, Leverkusen, DE) and 1.0 g 2,6-di-*tert.*-butylmethyl phenole were added and the mixture was heated to 60°C. Subsequently,
- 20 639.84 g of Tone M100[®] (Dow, Schwalbach, DE) were added dropwise at a rate such that the temperature did not exceed 70 °C. Stirring was continued until the NCO-content dropped below 0.1 %. This gave a colorless resin with a NCO-content of 0.08 % and a viscosity of 9050 mPas (23 °C).

Preparation of the urethane acrylate binder AG

- 25 A 2000-ml four-necked glass flask with reflux condenser, heatable oil bath, mechanical stirrer, air traversal (2 l/h), internal thermometer and dropping funnel was charged with 153.22 g of hexamethylene diisocyanate (Desmodur[®] H, Bayer Material Science AG, Leverkusen, DE). 0.50 g of dibutyltin dilaurate (Desmorapid[®] Z, Bayer MaterialScience AG, Leverkusen, DE) and 1.0g 2,6-di-*tert.*-butylmethyl phenole were added and the mixture was heated to 60 °C. Subsequently,
- 30 532.95 g of Oxyester T1136[®] (Degussa AG, Marl, DE) and then 313.73 g of Tone M100[®] (Dow, Schwalbach, DE) were added dropwise at a rate such that the temperature did not exceed 70 °C. Stirring was continued until the NCO-content dropped below 0.1 %. This gave a colorless resin with a NCO-content of 0 % and a viscosity of more than 500000 mPas (23 °C).

Reactive thinner B)

Reactive thinner BA is dipropylene glycol diacrylate (comparative).

Reactive thinner BB is Laromer[®] POEA, BASF SE, Ludwigshafen, DE (phenoxyethylacrylate).

5 Reactive thinner BC is OTA480[®], Cyttec Surface Specialities, SA/NV (propoxylated glycerol triacrylate (comparative).

Reactive thinner BD is Photomer[®] 4039, Cognis, Deutschland GmbH (phenol-(2,5)ethoxylate acrylate).

Reactive thinner BE is Photomer[®] 4050, Cognis, Deutschland GmbH (polyethylene glycol 200 diacrylate) (comparative).

10 Reactive thinner BF is Sartomer[®] SR-833 S, Sartomer div. of Cray Valley, F (tricyclodecanedi-methanol diacrylate) (comparative).

Reactive thinner BG is hexanediol diacrylate (comparative)

Reactive thinner BH is Sartomer[®] SR 256, Sartomer div. of Cray Valley, F (2-(2-ethoxyethoxy)ethyl acrylate).

15 Reactive thinner BI is Laromer[®] TBCH BASF SE, Ludwigshafen, DE, (4-t-butylcyclohexyl acrylate).

Photoinitiator C)

20 Photoinitiator CA is Darocur[®] 1173, Ciba Lampertheim, DE (1-phenyl-2-hydroxy-2-methyl-1-propane-1-one).

Preparation of the bonder formulation

25 All bonder materials depicted in Table 3 are formulated to such a ratio between binder A) and reactive thinner B) that the viscosity at 25 °C was about 480 to 520 mPas. Mixing of the bonder components was done according to procedures known to persons skilled in this art.

Description of the substrate molding process

Disk substrates (information substrate and dummy substrate) having a diameter of 120 mm and a thickness of 0.60 mm are injection molded from Makrolon® OD2015 from Bayer Material-
 Science AG using a Singulus E Mould DVD-R injection molding machines attached to a Singulus Streamline II DVD-R, equipped with ST-moulds from Axxicon moulds and a DVD+R or
 5 DVD-R stamper was used. Following molding parameters were used, see Table 2:

Table 2: Molding parameters according to disc molding process

T _{Cyl}	T _{Mold}	Clamp Force	Holding Time	Cooling Time	Injection Speed
(°C)	(°C)	(to)	(s)	(s)	(cm ³ /s)
360	123	32	0.5	2.3	220

T_{Cyl} is the melt temperature and T_{Mold} is the mold temperature, whereas the left value represents the mirror side of the mold and the right value the stamper side of the mold.

10 Description of the Production of the Dye and the Reflective Layer

DVD Recordable discs use an organic dye as recording layer by means of spin coating process. A fresh dye solution has been prepared by solubilizing the dye powder in tetrafluoropropanol as organic solvent. After the molding process, all substrates are transferred to temperate controlled conveyer to get efficient cooling of both sides of the substrate, ready for further dye coating
 15 process. The inline dye coating units are equipped with several automated dye dosing stations for the application of the precise controlled dye layer onto each grooved substrate. The spin coating process shall be strictly under a climate controlled environment. To remove residual solvent from the condensed dye film, the dye coated substrates are fed into the drying oven with infrared lamps.

20 For the deposition of a highly reflective metal layer on the top of the dye coated substrates, the high rate sputter module is used after drying process. The most common material is silver, or a silver alloy. A powerful and optimized metallizer can offer uniform sputtering throughout the target lifetime. Remove residual dye on the edge of the dye coated and metallized layer 0 substrates by dosing the diacetonaclcohol solvent after metallizing.

25 In this case the downstream process equipment of the above mentioned Singulus Streamline II DVD-R machine was used.

Description of the DVD+R (SL) inline bonding process

Single layer DVD+R discs were produced with the above substrates and the bonder formulations mentioned in table 4 using a Singulus Streamline II DVD-R machine. The bonding process is described as follows:

- 5 The metallized and edge-cleaned layer 0 substrate was transferred to the bonding unit, where the bonder material is dosed at a disc by spin coating bonding process. The dosage amount was 0.7 g. Afterwards another blank layer 1 substrate was placed on top. The bonder was distributed between the two substrates with optimized spin process. The bonder layer thickness achieved by this procedure was about 45µm. The bonder layer thickness was measured by Basler Scanner
10 REF 100.

The discs are exposed to ultraviolet radiation in the UV-curing station, to cure the bonding material by means of a radical polymerization reaction. The basic power of the UV lamp was 1.0 kW; Max. power of the UV lamp was 3.0 KW; Exposure time was 1.0 sec.

15 **Description of the manual bonding process**

600 µm polycarbonate disc / bonder / 600 µm polycarbonate disc sandwiches were fabricated as described below:

- A 600 µm thick polycarbonate disc substrate was put on the spin coater (Karl Suess Model RC 8) chuck. The bonder was applied with a pressurized dosage system (GLT Model ETD 2000
20 XL) at a disc radius of 35 mm. The spin speed was 200 RPM, the dosage time was 1.75 s. The syringe needle diameter used in the dosage system was 1.35 mm. The dosage pressure was adjusted to roughly 1.8 bar, so that a full circle of bonder material was dispersed on the substrate. A second 600 µm thick substrate was placed on top of the first substrate coated with the bonder. The bonder was distributed between the two substrate by spinning the sandwich structure at
25 1500 RPM for 10 second.

- Subsequent crosslinking of the bonder was carried out on a UV exposure apparatus. The sandwich structure of the two substrates and the bonder in between is placed on a conveyor belt, which is moved past the UV light source (Hg lamp, 80 W) at a speed of about 1 m/minute. A radiation dosage of about 5 J/cm² is applied to the sandwich structure to cure the bonder material and from the disc.
30

The bonder layer thickness achieved by this procedure was about 60 μm . The bonder layer thickness was measured by subtraction of the thicknesses of the two substrates from the total thickness of the bonded disc. All thicknesses were measured with a micrometer screw.

- 5 From these manually bonded discs and also from the produced DVD+R (SL) discs, samples were cut to examine the oscillation behaviour of the disc with a laser Doppler vibrometer. Especially at the stated test bar geometry modal oscillation occurs at 270 Hz. The damping related Q values at 270 Hz were extracted from the data for the samples under test. Q is $1/(3 \cdot \tan(\delta))$, whereas $\tan(\delta)$ is the mechanical damping. The results of these measurements are depicted in
- 10 Table 3 below.

Table 3: Composition of the bonder materials and the Q values at 270 Hz of the respective DVD+R disc that were bonded manually.

Example	Binder A	Weight fraction	Reactive thinner B	Weight fraction	Initiator C	Weight fraction	Q@270 Hz
1	AA	0.563	BB	0.408	CA	0.029	84
2	AA	0.485	BD	0.486	CA	0.029	68
3	AA	0.679	BH	0.291	CA	0.030	94
6	AD	0.728	BH	0.243	CA	0.029	66
7	AE	0.631	BH	0.340	CA	0.029	99
8	AF	0.728	BH	0.243	CA	0.029	128
9	AG	0.558	BH	0.413	CA	0.029	127
Comparative Example	Binder	Weight fraction	Reactive thinner	Weight fraction	Initiator	Weight fraction	Q@270 Hz
1	AA	0.561	BA	0.410	CA	0.029	258
2	AA	0.291	BC	0.680	CA	0.029	266
3	AA	0.515	BE	0.456	CA	0.029	196
4	AA	0.243	BF	0.728	CA	0.029	290
5	AA	0.602	BG	0.369	CA	0.029	255
6	DIC [®] 6830						227
7	Sony [®] SK 6500						199
16	AA	0.483	BH	0.146	CA	0.029	163
	AH	0.196	BG	0.146			

As can be seen from Table 3, the bonder materials according to the present invention improve the damping at 270 Hz of the disc at least by a factor of 1.53 compared to discs made with actual commercially available bonder materials like DIC[®] 6830 or Sony[®] SK 6500 (examples 6-7) and with bonders comprising diacrylates as reactive thinners (examples 1 to 5 and 16). Sony[®] SK 6500 is a special acrylate based spin coatable and UV-curable DVD bonder from Sony Chemical & Information Device Corporation Japan, not containing the inventive bonder. DIC[®] 6830 is a special acrylate based spin coatable and UV-curable DVD bonder from DIC Corporation Japan, not containing the inventive bonder material.

To proof the relation of the higher damping of the disc with improved tracking (TE) and focusing (FE) performance DVD+R (SL) disc were produced utilizing bonder materials from example 3 and comparative examples 6 and 7. Details how TE and FE were measured are described in section 9, subsection TE and FE signal evaluation. The results of these tests are summarized in Table 4 below.

Table 4: TE/FE signals at 16X from DVD+R (SL)'s made with dedicated bonders

Example	Bonder-material	Substrate polycarbonate	Max. radial deviation (°)	Max. tangential deviation (°)	TE	FE	Q@270 Hz
10 Plextools	Example 3	Makrolon [®] OD 2015	0.76	0.15	32	16	94
10 QScan	Example 3	Makrolon [®] OD 2015	0.76	0.15	251	89	94
Comparative Example	Bonder-material	Substrate polycarbonate	Max. radial deviation (°)	Max. tangential deviation (°)	TE	FE	Q@270 Hz
17 Plextools	DIC [®] 6830	Makrolon [®] OD 2015	0.78	0.22	41	22	227
17 QScan	DIC [®] 6830	Makrolon [®] OD 2015	0.78	0.22	291	109	227
18 Plextools	Sony [®] SK 6500	Makrolon [®] OD 2015	0.51	0.08	48	20	199

As can be seen clearly from the results above the bonder material of the present invention significantly improves TE and FE signals of the final DVD discs.

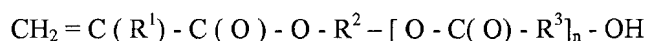
CLAIMS:

1. A DVD comprising discs made from polycarbonate comprising a UV curable bonder material consisting of

A) an urethane acrylate oligomer as binder, which is the reaction product of

5 i) one or more polyisocyanates and

ii) one or more hydroxyfunctional lactone ester acrylates having a number average molecular weight of from 200 g/mol to 2000 g/mol and having the formula:



10 wherein , n is an integer from 1 to 5, R¹ is hydrogen or methyl, R² represents an alkylene group or substituted alkylene group having from 2 to 10 carbon atoms and R³ represents a straight or branched chain alkylene group of from 3 to 8 carbon atoms, and which may be substituted with one or more alkyl groups having from 1 to 12 carbon atoms,

B) a reactive thinner, comprising monofunctional (meth)acrylates

15 C) a photoinitiator and

D) optional additives.

2. A DVD comprising discs made from polycarbonate according to claim 1 wherein the polyisocyanate component i) of the urethane acrylate A) is an oligomer of hexamethylenediisocyanate and/or dicyclohexylmethanediisocyanate.

20 3. A DVD comprising discs made from polycarbonate according to claim 1 to 2 wherein the hydroxylcontaining component ii) of the urethane acrylate A) is the reaction product of hydroxyethylacrylate, hydroxypropylacrylate or hydroxybutylacrylate with ε-caprolactone.

4. A DVD comprising discs made from polycarbonate according to claim 1 to 3 wherein component B) is selected from the group comprising alkoxyated alkyl (meth)acrylates, monofunctional esters of (meth)acrylic acid and alkoxyated phenoles, alkyl (meth)acrylates, cycloaliphatic (meth)acrylates and heterocyclic (meth)acrylates.

25

5. A DVD comprising discs made from polycarbonate according to claim 1 to 4 wherein component B) is selected from the group comprising C1- to C4- alkoxyated C1- to C4- al-

kyl (meth)acrylates, monofunctional esters of (meth)acrylic acid and C1- to C4- alkoxy-
lated phenols, C1- to C10- alkyl (meth)acrylates, C4- to C10- cycloaliphatic
(meth)acrylates and C4- to C6 heterocyclic (meth)acrylates.

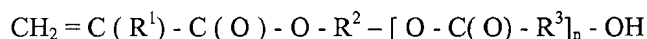
6. A DVD comprising discs made from polycarbonate according to claim 1 to 5 wherein
5 component B) is selected from the group consisting of phenoxy-ethylacrylate, phenole-
(2,5-diethoxy) acrylate, 2-(2-ethoxyethoxy)ethylacrylate, isobornyl acrylate and tetrahy-
drofurfuryl acrylate.

7. Use of a UV-curable bonder material consisting of

A) an urethane acrylate oligomer as binder, which is the reaction product of

- 10 i) one or more polyisocyanates and

ii) one or more hydroxyfunctional lactone ester acrylates having a number average mo-
lecular weight of from 200 g/mol to 2000 g/mol and having the formula:



- 15 wherein , n is an integer from 1 to 5, R¹ is hydrogen or methyl, R² represents an al-
kylene group or substituted alkylene group having from 2 to 10 carbon atoms and R³
represents a straight or branched chain alkylene group of from 3 to 8 carbon atoms, and
which may be substituted with one or more alkyl groups having from 1 to 12 carbon at-
oms,

B) a reactive thinner, comprising monofunctional acrylates

- 20 C) a photoinitiator and

D) optional additives,

as bonder material for the production of DVDs.

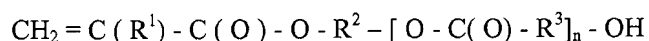
8. Use of a UV-curable bonder material according to Claim 7 for the production of DVDs,
showing a Q value of less than 130 measured at 270 Hz.

- 25 9. Process for the production of DVDs comprising discs made from polycarbonate whereby in
a step 1 on a first polycarbonate disc substrate, comprising optionally dye and reflective
layers, a UV-curable bonder composition consisting of

A) an urethane acrylate oligomer as binder, which is the reaction product of

i) one or more polyisocyanates and

ii) one or more hydroxyfunctional lactone ester acrylates having a number average molecular weight of from 200 g/mol to 2000 g/mol and having the formula:



5 wherein , n is an integer from 1 to 5, R¹ is hydrogen or methyl, R² represents an alkylene group or substituted alkylene group having from 2 to 10 carbon atoms and R³ represents a straight or branched chain alkylene group of from 3 to 8 carbon atoms, and which may be substituted with one or more alkyl groups having from 1 to 12 carbon atoms,

10 B) a reactive thinner, comprising monofunctional acrylates

C) a photoinitiator and

D) optional additives,

is applied and

15 in step 2 a second polycarbonate disc substrate is placed on top of the coated substrate resulting from step 1 and in step 3 the resulting product from step 2 is cured by UV-radiation.

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25

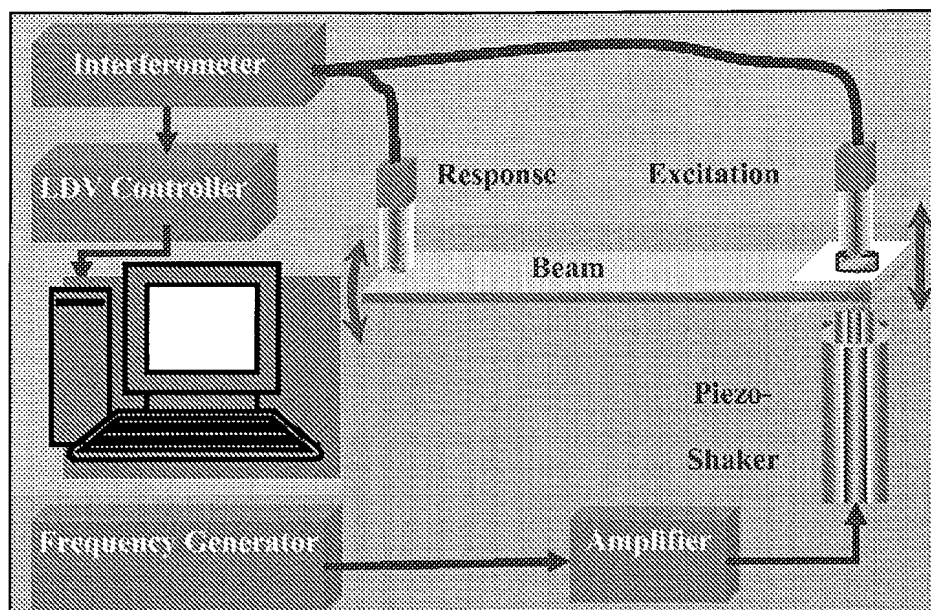


Figure 1: Setup of the Laser-Doppler-Vibrometer Method

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2011/051063

A. CLASSIFICATION OF SUBJECT MATTER
INV. G11B7/246
ADD.

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
G11B

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

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 00/75253 A1 (3M INNOVATIVE PROPERTIES CO [US]; FUJII SANA [JP]) 14 December 2000 (2000-12-14) example comparative 5 -----	1,7,9
X	US 6 335 382 B1 (IIDA TAKAFUMI [JP]) 1 January 2002 (2002-01-01) claims 10,12; example 3 column 3, line 53 - column 5, line 14 -----	1-7,9
A	EP 1 671 995 A1 (BAYER MATERIALSCIENCE LLC [US]) 21 June 2006 (2006-06-21) claims 1,8,17 -----	1
A	WO 2008/125199 A1 (BAYER MATERIALSCIENCE AG [DE]; INPHASE TECH INC [US]; STOECKEL NICOLAS) 23 October 2008 (2008-10-23) example 4 -----	1

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

☒ See patent family annex.

* Special categories of cited documents :

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

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

8 April 2011

Date of mailing of the international search report

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/EP2011/051063

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
WO 0075253	A1	14-12-2000	EP 1183316 A1 JP 2000345111 A	06-03-2002 12-12-2000
US 6335382	B1	01-01-2002	WO 2004074391 A1	02-09-2004
EP 1671995	A1	21-06-2006	CA 2529181 A1 CN 1800271 A JP 2006169527 A KR 20060067869 A MX PA05013778 A US 2006128923 A1	15-06-2006 12-07-2006 29-06-2006 20-06-2006 19-06-2006 15-06-2006
WO 2008125199	A1	23-10-2008	CA 2683882 A1 CN 101668782 A EP 2137220 A1 JP 2010524036 T KR 20100015471 A US 2008312403 A1	23-10-2008 10-03-2010 30-12-2009 15-07-2010 12-02-2010 18-12-2008



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W·菲谢尔 M·路德维希 王良文

权利要求书 2 页 说明书 15 页 附图 1 页

(54) 发明名称

高速 DVD

(57) 摘要

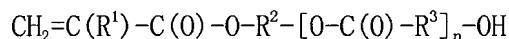
本发明涉及由聚碳酸酯和特殊 UV 可固化粘合剂材料制成的 DVD, 所述粘合剂材料由下列组成: A) 作为粘合剂的氨基甲酸乙酯丙烯酸酯低聚物, 所述低聚物为下列的反应产物: i) 一种或多种聚异氰酸酯, 和 ii) 一种或多种羟基官能内酯(甲基)丙烯酸酯, 其数均分子量为 200g/mol 至 2000g/mol, 并且具有下式: $\text{CH}_2=\text{C}(\text{R}^1)-\text{C}(\text{O})-\text{O}-\text{R}^2-[\text{O}-\text{C}(\text{O})-\text{R}^3]_n-\text{OH}$, 其中 n 为 1 至 5 的整数, R^1 为氢或甲基, R^2 表示具有 2 至 10 个碳原子的亚烷基或经取代的亚烷基, 并且 R^3 表示可用一个或多个具有 1 至 12 个碳原子的烷基取代的 3 至 8 个碳原子的直链或支链亚烷基; B) 包含单官能丙烯酸酯和 / 或 (甲基) 丙烯酸酯的活性稀释剂; C) 光引发剂; 和 D) 任选的添加剂, 所述 DVD 提供在高数据传输速率改进的性能。

1. 一种包含由聚碳酸酯制成的光盘的 DVD, 所述 DVD 包含由下列组成的 UV 可固化粘合剂材料:

A) 作为粘合剂的氨基甲酸乙酯丙烯酸酯低聚物, 所述低聚物为下列的反应产物:

i) 一种或多种聚异氰酸酯, 和

ii) 一种或多种羟基官能内酯丙烯酸酯, 其数均分子量为 200g/mol 至 2000g/mol, 并且具有下式:



其中 n 为 1 至 5 的整数, R^1 为氢或甲基, R^2 表示具有 2 至 10 个碳原子的亚烷基或经取代的亚烷基, 并且 R^3 表示可用一个或多个具有 1 至 12 个碳原子的烷基取代的 3 至 8 个碳原子的直链或支链亚烷基;

B) 包含单官能(甲基)丙烯酸酯的活性稀释剂;

C) 光引发剂; 和

D) 任选的添加剂。

2. 权利要求 1 的包含由聚碳酸酯制成的光盘的 DVD, 其中氨基甲酸乙酯丙烯酸酯 A) 的聚异氰酸酯组分 i) 为六亚甲基二异氰酸酯和 / 或二环己基甲烷二异氰酸酯的低聚物。

3. 权利要求 1 至 2 的包含由聚碳酸酯制成的光盘的 DVD, 其中氨基甲酸乙酯丙烯酸酯 A) 的含羟基组分 ii) 为丙烯酸羟基乙酯、丙烯酸羟基丙酯或丙烯酸羟基丁酯与 ϵ -己内酯的反应产物。

4. 权利要求 1 至 3 的包含由聚碳酸酯制成的光盘的 DVD, 其中组分 B) 选自(甲基)丙烯酸烷氧基化烷基酯、(甲基)丙烯酸和烷氧基化酚的单官能酯、(甲基)丙烯酸烷基酯、(甲基)丙烯酸脂环酯和(甲基)丙烯酸杂环酯。

5. 权利要求 1 至 4 的包含由聚碳酸酯制成的光盘的 DVD, 其中组分 B) 选自(甲基)丙烯酸 C1- 至 C4- 烷氧基化 C1- 至 C4- 烷基酯、(甲基)丙烯酸和 C1- 至 C4- 烷氧基化酚的单官能酯、(甲基)丙烯酸 C1- 至 C10- 烷基酯、(甲基)丙烯酸 C4- 至 C10- 脂环酯和(甲基)丙烯酸 C4- 至 C6- 杂环酯。

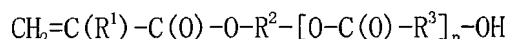
6. 权利要求 1 至 5 的包含由聚碳酸酯制成的光盘的 DVD, 其中组分 B) 选自丙烯酸苯氧基乙酯、丙烯酸苯酚-(2,5-二乙氧基)酯、丙烯酸 2-(2-乙氧基乙氧基)乙酯、丙烯酸异冰片酯和丙烯酸四氢糠酯。

7. UV 可固化粘合剂材料用作制造 DVD 的粘合剂材料的用途, 所述粘合剂材料由下列组成:

A) 作为粘合剂的氨基甲酸乙酯丙烯酸酯低聚物, 所述低聚物为下列的反应产物:

i) 一种或多种聚异氰酸酯, 和

ii) 一种或多种羟基官能内酯丙烯酸酯, 其数均分子量为 200g/mol 至 2000g/mol, 并且具有下式:



其中 n 为 1 至 5 的整数, R^1 为氢或甲基, R^2 表示具有 2 至 10 个碳原子的亚烷基或经取代的亚烷基, 并且 R^3 表示可用一个或多个具有 1 至 12 个碳原子的烷基取代的 3 至 8 个碳原子的直链或支链亚烷基;

B) 包含单官能丙烯酸酯的活性稀释剂;

- C) 光引发剂 ;和
- D) 任选的添加剂。

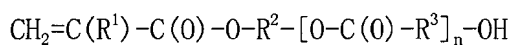
8. 权利要求 7 的 UV 可固化粘合剂材料用于制造 DVD 的用途,所述 DVD 显示在 270Hz 测量的小于 130 的 Q 值。

9. 一种制造包含由聚碳酸酯制成的光盘的 DVD 的方法,其中在步骤 1,在包含任选的染料和反射层的第一聚碳酸酯光盘基片上涂覆 UV 可固化粘合剂组合物,所述组合物由下列组成:

A) 作为粘合剂的氨基甲酸乙酯丙烯酸酯低聚物,所述低聚物为下列的反应产物:

i) 一种或多种聚异氰酸酯,和

ii) 一种或多种羟基官能内酯丙烯酸酯,其数均分子量为 200g/mol 至 2000g/mol,并且具有下式:



其中 n 为 1 至 5 的整数, R^1 为氢或甲基, R^2 表示具有 2 至 10 个碳原子的亚烷基或经取代的亚烷基,并且 R^3 表示可用一个或多个具有 1 至 12 个碳原子的烷基取代的 3 至 8 个碳原子的直链或支链亚烷基;

B) 包含单官能丙烯酸酯的活性稀释剂;

C) 光引发剂 ;和

D) 任选的添加剂,

并且

在步骤 2,在从步骤 1 得到的经涂布的基片上放置第二聚碳酸酯光盘基片,在步骤 3,通过 UV 辐射固化来自步骤 2 的所得产物。

高速 DVD

[0001] 本发明描述特殊 UV 粘合剂用于制造 DVD (数字多用光盘) 的用途, 所述粘合剂促进在高数据传输速率下改进聚焦和跟踪性能。

[0002] 光学数据存储发展的主要特征是每张光盘的存储容量从一代向下一代增加。这可以例如通过以下作为例证, 在压缩光盘 (CD) 格式中每张盘 0.7GB 容量, 单层数字多用光盘 (DVD) 4.7GB 容量, 单层蓝光光盘 (BD) 25GB 容量。如在 DVD 和 BD 的情况可见的那样, 那些容量可通过引入例如双层光盘或具有甚至更多层或双侧可存取光盘来增加。

[0003] 另外的重要一点是在光盘的读出和 / 或记录期间的数据传输速率。由于依次读出和 / 或记录数据, 增加存储密度导致增加整盘的读出和 / 或记录时间, 尽管间隔 (pit) 或记录标记从一代到下一代变小。传输速率的不足抵消了每种格式的 1X 记录和读出的基准速度从一代到下一代增加。为了增加一种格式内的数据传输速率, 增加光盘的旋转速度促进高速记录和读出。

[0004] 对于 CD 格式, 已开发能够达到与 1X 基准速度相比 52X 数据传输速率的驱动器和光盘。由于对于 0.7GB 容量光盘, 线基准速度为 1.2m/s, 对于 52X 速度, 旋转速度为约 10500 转 / 分钟 (RPM)。

[0005] 对于 DVD 格式, 已开发能够达到与 1X 基准速度相比最多 22X 数据传输速率的驱动器和光盘。对于 22X 速度, 旋转速度为约 12830RPM。高旋转速度显著减少记录时间, 可从以下表 1 看到。

[0006] 表 1: 在第一驱动器和媒体模型中具有 1X 的 DVD 的写速度, 即 1350kB/s。更近来的模型在 18X 或 20X 工作, 这意味着最大数据传输速率为 18 或 20 倍高。应注意, 对于 CD 驱动器, 1X 是指 150kB/s, 为 1X DVD 的约 1/9 慢

DVD驱动速度				
驱动速度	最大数据传输速率		~写时间(min)	
	(Mbit/s)	(MB/s)	单层	双层
1X	10.55	1.35	61	107
2X	21.09	2.70	30	54
2.4X	27.43	3.51	24	42
4X	42.19	5.40	15	27
6X	63.30	8.10	11	18
8X	84.38	10.80	8	14
12X	126.60	16.20	6	11
16X	168.75	21.60	4	7
18X	189.90	24.30	3	5
20X	211.00	27.00	3	4

[0007] 对于 BD 格式,存在支持与 1X 基准速度相比最高 8X 数据传输速率的光盘和驱动器。由于对于 25GB/ 层容量光盘,1X 基准速度的线速度为 4.92m/s,对于 8X 速度,旋转速度为约 6480RPM。在实验室中,已证明 12X 记录速度,它相当于 9720RPM 光盘旋转速度(在 Takamatsu, Japan 举行的 ISOM 2006(International Symposium of Optical Memory) (国际光存储研讨会)的会议记录,“Up to 12X BD-R(Cu:Si) Recording”(最高 12X BD-R(Cu:Si)记录), R. G. J. A. Hesens, J. H. G. Jaegers, A. P. G. E. Janssen, J. Rijpers, P. R. V. Sonnevile, J. J. H. B. Schleipen, Philips Research Laboratories, High Tech Campus 34, 5656 AE Eindhoven, The Netherlands)。

[0008] 在这种高旋转速度,激光的跟踪和聚焦对高品质记录和读出数据变得极其重要。在此情况下,可能发生光盘读出器或写入器减小光盘旋转速度,以便以较低错误率得到较佳数据信号品质。因而不能得到高旋转速度。

[0009] 例如,DVD 写入器生产商通常在对记录进行功率校准之前检查高速(例如 16X)下的跟踪误差(TE)和聚焦误差(FE)信号的品质。如果 TE 或 FE 高于写入器具体限制,就以低得多的速度进行记录。检查这些 TE 和 FE 品质的免费软件工具的实例为用于 Plextor 写入器的 Plectools,和对大多数 BenQ 和 Philips 写入器运行的 Qscan。

[0010] 由于对于光学拾取系统,如果光盘在下面旋转,其光轴与数据轨道的工作距离和对准随时间改变,因此,适合的跟踪和聚焦强烈关系到光盘表面的平坦度。那意味着光盘的低切向偏差是重要的,这意味着对于拾取头的上述性质的周期和同步变化。对低切向和轴向偏差的高需求使高速能力 DVD 生产的处理窗口变窄,且对光盘生产商意味着额外负担。

[0011] 而且模态振动可得到在高旋转速度相对于焦距显著的振幅。由于跟踪和聚焦伺服

系统有带宽限制,并且不能足够快地遵循大和低频失真,这些模态振动也产生工作距离和对准的非同步瞬时变化,这使跟踪和聚焦在高旋转速度困难。模态振动的特征是其模态频率 f_n ,它与光盘的几何形状和按照 f_n 与 $(E/\rho)^{0.5}$ 成比例的杨氏模量 E 和质量密度的比率相关(见(3))。通过 $Q=3/\tan \sigma$,品质因子 Q (见公式(4))与 $\tan \sigma$ 相关。在此意义上,象 $\tan \sigma$ 一样, Q 可用作阻尼的度量。低 Q 意味着高阻尼,因为 $\tan \sigma$ 高。一般 E 和 Q 显示对频率 f 的不同依赖性。由聚碳酸酯制成的 12cm 直径和 1.2mm 厚光盘的最低频率模态振动的组合在 130Hz 至 170Hz 范围 [S. Subramanian, R. Hariharan, B. Landa, T. P. Foist, Optical Data Storage(光学数据存储)2000, SPIE 会议记录 Vol. 4090 (2000), pp 323 ff, WO 0048172A2, 比较实施例 1]。下一组模态振动发生在约 280Hz。由于在 16X 的旋转速度为约 155Hz,这种模态振动可容易地在光盘操作期间激发。

[0012] WO 0048172A2 公开通过控制硬挺度和阻尼来抑制模态振动对高密度数据存储(>5Gbit/in² 面密度 >DVD 密度)介质的影响的解决方法。然而,由于信息层在该介质的第一表面,可通过不透明基片材料满足硬挺度和阻尼要求。公开的解决方法利用在经济 DVD 生产中不能使用的夹层注模方法。

[0013] EP 1 158 024 A1 描述一种振动-阻尼热塑性树脂组合物,所述组合物包含 a) 50-90 重量%的非晶热塑性树脂,所述树脂具有 0.01 至 0.04 的损失 $\tan \sigma$ 和在负荷下不低于 120°C 的挠曲温度;和 b) 50 至 10 重量%的甲基丙烯酸甲酯树脂,其中从所述组合物模压的制品具有特定的物理性质。公开的解决方法实现提高的阻尼,然而使用特殊聚碳酸酯与甲基丙烯酸甲酯的聚合物掺合物。公开的解决方法的光学透明度对于 DVD 应用不够好。

[0014] WO 2006046864A1 公开用从二醇、二异氰酸酯和包含羟基的(甲基)丙烯酸酯得到的氨基甲酸乙酯丙烯酸酯与环氧二丙烯酸酯、单官能和二官能丙烯酸酯组合作为光盘的粘合剂。然而,已证明二官能丙烯酸酯以及环氧丙烯酸酯不适合用于高旋转速度的 DVD 光盘的粘合剂(参见比较实施例 16)。

[0015] CN 1456621A 公开适用于 DVD 的 UV 可固化涂层。这些 UV 可固化涂层包含氨基甲酸酯-丙烯酸酯作为活性低聚物、单官能和二官能(甲基)丙烯酸酯、光引发剂和光学添加剂。

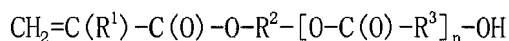
[0016] 因此,仍存在能够在高旋转速度提供改进的跟踪和聚焦操作的 DVD 光盘的必要性。使用标准 DVD 粘合剂材料已利用紧窄的生产余地来保持 TE 和 FE 信号在高记录速度下在可接受的限度内。通常,由于不良的跟踪或聚焦性能,DVD 写入器不接受光盘用于 16X 或甚至更高的 X 记录。

[0017] 因此,提供在高旋转速度相对于跟踪和聚焦显示较宽余地的 DVD 是一项任务。

[0018] 已意外地发现,使用特殊 UV 可固化粘合剂可解决上述问题。本发明涉及用聚碳酸酯与特殊粘合剂材料和任选的附加层制成的 DVD。

[0019] UV 可固化粘合剂材料一般为由下列组成的可旋涂制剂:

- A) 作为粘合剂的氨基甲酸乙酯丙烯酸酯低聚物,所述低聚物为下列的反应产物:
 - i) 一种或多种聚异氰酸酯,和
 - ii) 一种或多种羟基官能内酯丙烯酸酯,其数均分子量为 200g/mol 至 2000g/mol,并且具有下式:



其中 n 为 1 至 5 的整数, R^1 为氢或甲基, R^2 表示具有 2 至 10 个碳原子的亚烷基或经取代的亚烷基, 并且 R^3 表示可用一个或多个具有 1 至 12 个碳原子的烷基取代的 3 至 8 个碳原子的直链或支链亚烷基;

B) 包含单官能(甲基)丙烯酸酯的活性稀释剂;

C) 光引发剂;和

D) 任选的添加剂。

[0020] 另外发现, 这种特殊 UV 可固化粘合剂在用于 DVD 生产时, 得到具有在 270Hz 频率测量的小于 130 的 Q 因数的光盘。

[0021] 简短地讲, 用能够在室温在约 200Hz 至 300Hz 频率水平改进阻尼的粘合剂制成的 DVD 光盘提供在 16X 或以上的记录速度显著改进的跟踪和聚焦性能。

[0022] 在本说明书的上下文中, “(甲基)丙烯酸酯” 涉及相应的丙烯酸酯或(甲基)丙烯酸酯官能或两者的混合物。

[0023] 氨基甲酸乙酯丙烯酸酯低聚物 A) 可根据 EP-A 1 650 241 或 EP-A 1 671 995 所述的方法制备。

[0024] 氨基甲酸乙酯丙烯酸酯低聚物 A) 中的聚异氰酸酯组分 (i) 包含:

● 选自芳族、芳脂族、脂族或脂环族聚异氰酸酯的聚异氰酸酯或这些聚异氰酸酯的混合物, 例如 1, 3- 环己烷二异氰酸酯、1- 甲基-2, 4- 二异氰酸酯基- 环己烷、1- 甲基-2, 6- 二异氰酸酯基- 环己烷、四亚甲基二异氰酸酯、4, 4'- 二异氰酸酯基二苯基甲烷、2, 4'- 二异氰酸酯基二苯基甲烷、2, 4- 二异氰酸酯基甲苯、2, 6- 二异氰酸酯基甲苯、 $\alpha, \alpha, \alpha, ' \alpha, ' \alpha$ - 四甲基- 间- 或- 对- 苯二亚甲基二异氰酸酯、1, 6- 六亚甲基二异氰酸酯、1- 异氰酸酯基-3, 3, 5- 三甲基-5- 异氰酸酯基甲基环己烷(异佛尔酮二异氰酸酯或 IPDI)、4, 4'- 二异氰酸酯基- 二环己基甲烷、4- 异氰酸酯基甲基-1, 8- 辛烷二异氰酸酯(三异氰酸酯基壬烷, TIN)(EP-A 928 799) 及其混合物。所述聚异氰酸酯与缩二脲、碳二亚胺、异氰脲酸酯、脲基甲酸酯、亚氨基噁二嗪二酮和 / 或缩脲二酮基的均聚物或低聚物、它们相互之间的混合物和与上述聚异氰酸酯的混合物也适用。优选 1, 6- 六亚甲基二异氰酸酯、1- 异氰酸酯基-3, 3, 5- 三甲基-5- 异氰酸酯基甲基环己烷(异佛尔酮二异氰酸酯或 IPDI) 和 4, 4'- 二异氰酸酯基- 二环己基甲烷和它们相互之间的混合物。也优选 1- 异氰酸酯基-3, 3, 5- 三甲基-5- 异氰酸酯基甲基环己烷(异佛尔酮二异氰酸酯或 IPDI) 和 4, 4'- 二异氰酸酯基- 二环己基甲烷与缩二脲、碳二亚胺、异氰脲酸酯、脲基甲酸酯、亚氨基噁二嗪二酮和 / 或缩脲二酮基的均聚物或低聚物、它们相互之间的混合物和与上述优选的聚异氰酸酯的混合物。

[0025] ● 由聚酯二醇和 / 或聚醚二醇和六亚甲基二异氰酸酯制成的六亚甲基二异氰酸酯的预聚物;

● 由聚酯二醇和 / 或聚醚二醇和二环己基甲烷二异氰酸酯制成的二环己基甲烷二异氰酸酯的预聚物。

[0026] 六亚甲基二异氰酸酯和 / 或二环己基甲烷二异氰酸酯的低聚物特别优选作为聚异氰酸酯组分 i)。

[0027] 丙烯酸羟基乙酯、丙烯酸羟基丙酯或丙烯酸羟基丁酯与 ϵ - 己内酯的反应产物最优选作为氨基甲酸乙酯丙烯酸酯低聚物 A) 中的羟基官能内酯丙烯酸酯 ii)。

[0028] 活性稀释剂 B) 包含单官能 (甲基) 丙烯酸酯。

[0029] 优选的化合物 B) 选自 (甲基) 丙烯酸烷氧基化烷基酯、(甲基) 丙烯酸和 C1 至 C10- 烷氧基化酚的单官能酯、(甲基) 丙烯酸烷基酯、(甲基) 丙烯酸脂环酯和 (甲基) 丙烯酸杂环酯。

[0030] 特别优选的化合物 B) 包含选自 (甲基) 丙烯酸 C1- 至 C4- 烷氧基化 C1- 至 C4- 烷基酯、(甲基) 丙烯酸和 C1- 至 C4- 烷氧基化酚的单官能酯、(甲基) 丙烯酸 C1- 至 C10- 烷基酯、(甲基) 丙烯酸 C4- 至 C10- 脂环酯和 (甲基) 丙烯酸 C4- 至 C6- 杂环酯的化合物。

[0031] 最特别优选的化合物 B) 包含选自丙烯酸苯氧基乙酯、丙烯酸苯酚-(2,5-二乙氧基) 酯、丙烯酸 2-(2-乙氧基乙氧基) 乙酯、丙烯酸异冰片酯和丙烯酸四氢糠酯的化合物。

[0032] 典型的光引发剂 C) 包含羟基苯基酮, 例如 (1-羟基环己基) 苯基酮 (例如, Irgacure[®] 184, Ciba, Lampertheim, DE)、1-羟基二甲基苯基酮 (例如, Darocur[®] 1173, Ciba, Lampertheim, DE); 酰基氧化膦, 例如 2,4,6-三甲基-苯甲酰基-二苯基氧化膦 (例如, Darocur[®] TPO, Ciba, Lampertheim, DE)、苯基双-(2,4,6-三甲基苯甲酰基) 氧化膦 (例如, Irgacure[®] 819, Ciba, Lampertheim, DE); 苯基乙醛酸酯, 例如苯基乙醛酸 (甲基) 酯 (例如, Irgacure[®] MBF, Ciba, Lampertheim, DE) 和苄基二甲基缩酮 (例如, Irgacure[®] 651, Ciba, Lampertheim, DE)。

[0033] 在涂料和粘合剂技术中已知的添加剂、粘合剂和辅助物质 D) 可加到粘合剂材料中或与粘合剂材料组合。这些物质为流动和润湿添加剂、润滑添加剂、填料、纳米颗粒、光稳定颗粒、抗变黄添加剂、增稠剂和用于减小表面张力的添加剂。

[0034] 根据本发明, UV 固化粘合剂材料配制成分 A) 与 B) 之间的比率使得粘度在 25°C 为约 200 至 1000 mPas, 优选约 350 至 800 mPas, 特别优选约 450 至 650 mPas。

[0035] 本发明的 UV 固化粘合剂组合物中的光引发剂 C) 的含量为基于所有可聚合化合物的 1 至 10 重量%, 优选 2 至 8 重量%, 特别优选 3 至 6 重量%。

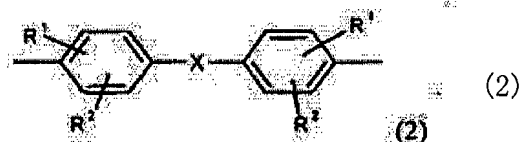
[0036] 基片材料的一般描述

聚碳酸酯树脂一般通过芳族二羟基化合物和碳酸酯前体的溶液聚合或熔体聚合来获得。如果由相应的聚碳酸酯制成的光盘满足高旋转速度试验的要求 (其中所述试验描述于说明书的试验部分) 和 / 或各自的聚碳酸酯满足以上相对溶液粘度范围, 任何芳族二羟基化合物都是可接受的。

[0037] 优选的芳族二羟基化合物为式 (1) 的化合物

HO-Z-OH (1)

其中 Z 表示式 (2) 的基团



其中

R¹ 和 R² 相互独立表示 H 或 C₁-C₆- 烷基, 优选 H 或 C₁-C₄- 烷基, 特别是氢或甲基; 并且

X 表示可通过 C₁-C₆- 烷基 (优选甲基或乙基) 取代的单键、C₁-C₆- 亚烷基、C₂-C₅- 烷叉基或 C₅-C₆- 环烷叉基, 其条件为如果 X 表示 3,3,5-三甲基环己叉基, R¹ 和 R² 表示氢。

[0038] 最优选 X 表示异丙叉基, R^1 和 R^2 表示氢。

[0039] 芳族二羟基化合物一般已知, 或者可根据一般已知的方法制备。聚碳酸酯的界面或熔体聚合也一般已知, 并且描述于很多公布中, 例如 WO 2004/063249 A1、WO 2001/05866 A1、WO 2000/105867、US-A 5, 340, 905、US 5, 097, 002、US-A 5, 717, 057。

[0040] 聚碳酸酯树脂可以为通过以上芳族二羟基化合物均聚得到的均聚碳酸酯或通过两种或更多种上述芳族二羟基化合物共聚得到的共聚碳酸酯。基于双酚 A 作为二酚单体的聚碳酸酯可包含基于制备聚碳酸酯所用二酚的量最高 20 重量 % (优选最高 10 重量 %, 更优选最高 5 重量 %) 的选自 1, 1- 双 (4- 羟基苯基) -3, 3, 5- 三甲基环己烷、 α , α' - 双 (4- 羟基苯基) 间 - 二异丙基苯或 4, 4'- 二羟基 - 联苯或 2- 苯基 -3, 3- 双 (4'- 羟基苯基) 邻苯二甲酰亚胺的另外的二酚单体。

[0041] 通过溶液方法反应通常为芳族二羟基化合物和光气之间的反应, 并且通常在酸偶联剂和有机溶剂存在下进行。作为酸偶联剂, 使用碱金属氢氧化物, 例如氢氧化钠或氢氧化钾; 或胺化合物, 例如吡啶。用卤化烃作为有机溶剂, 例如二氯甲烷或氯苯或这些烃的混合物。为了加速反应, 也可使用催化剂, 例如叔胺、季铵化合物或季磷化合物, 分别例如三乙胺、N- 乙基 - 哌啶、溴化四正丁基铵或溴化四正丁基磷。优选反应温度常规为 0 至 40°C, 反应时间为 10 秒至 5 小时, 并且在反应期间的 pH 不小于 9。反应可以为分批过程或连续过程。

[0042] 在聚合反应中, 通常也利用调节相对溶液粘度的封端剂。使用的这些封端剂可以为单官能酚。得到的聚碳酸酯树脂具有由基于单官能酚的基团封端的末端, 因而其热稳定性优于未如上述得到的聚碳酸酯树脂。单官能酚一般为酚或低碳烷基取代的酚, 例如苯酚、对 - 叔丁基苯酚、对 - 枯基苯酚、异辛基苯酚; 或长链烷基酚, 例如癸基苯酚、十二烷基苯酚、十六烷基苯酚、十八烷基苯酚、二十烷基苯酚、二十二烷基苯酚和三十烷基苯酚。优选的封端剂为苯酚、对叔丁基 - 苯酚、对枯基苯酚和 3- 正十五烷基苯酚 (CAS # 501-24-6)。

[0043] 封端剂以得到适合的相对溶液粘度的量引入。封端剂可单独与其他上述酚化合物组合使用。

[0044] 通过熔融方法反应通常为上述芳族二羟基化合物和碳酸酯之间的酯交换反应, 并且通过包括在惰性气体存在下在加热下混合芳族二羟基化合物和碳酸酯和蒸馏出产生的醇或酚的方法进行。虽然反应温度与例如产生的醇或酚的沸点不同, 但通常为 120 至 350°C。在后半反应期间, 反应系统减压到约 1.33×10^3 至 13.3 Pa, 以促进蒸馏产生的醇或酚。反应时间通常为 1 至 6 小时。

[0045] 在碳酸酯中, 有有时可经取代的酯, 例如 C6 至 C10 芳基或芳烷基或 C1-4 烷基, 具体为碳酸二苯酯、碳酸双 (氯苯基) 酯、碳酸二萘酯、碳酸双 (二苯基) 酯、碳酸二甲酯、碳酸二乙酯和碳酸二丁酯。其中碳酸二苯酯是最优选的。

[0046] 为了加速聚合, 也可使用聚合催化剂。可用常规用于酯化反应或酯交换反应的那些催化剂作为这些聚合催化剂, 例如碱金属化合物, 例如氢氧化钠、氢氧化钾或二元酚的钠或钾盐; 碱土金属化合物, 例如氢氧化钙、氢氧化钡或氢氧化镁; 含氮碱性化合物, 例如氢氧化四甲铵、氢氧化四乙铵、三甲胺或三乙胺; 碱金属或碱土金属的醇盐; 含磷碱性化合物, 例如四苯基磷酚盐; 或碱金属或碱土金属的有机酸盐; 锌化合物; 硼化合物; 铝化合物

物；硅化合物；锆化合物；有机锡化合物；铅化合物；钨化合物；铋化合物；锰化合物；钛化合物或钨化合物。这些催化剂可单独或组合使用。这些催化剂的用量优选为 1×10^{-8} 至 1×10^{-3} 当量，更优选 1×10^{-7} 至 5×10^{-4} 当量，相对于 1 摩尔二元酚作为起始材料。

[0047] 由于在聚合中的异构化反应，芳族聚碳酸酯树脂可在聚合物中含有三官能或更高官能芳族化合物或含有支化组分。三官能或更高官能芳族化合物的实例优选包括间苯三酚 (phlorogrucin)、2,3',4,5',6- 五羟基联苯 (phlorogrucide)；三酚，例如，4,6- 二甲基-2,4,6- 三-(4-羟基苯基)庚烯-2,2,4,6- 三甲基-2,4,6- 三(4-羟基苯基)庚烷、1,3,5- 三(4-羟基苯基)苯、1,1,1- 三(4-羟基苯基)乙烷、1,1,1- 三(3,5- 二甲基-4-羟基苯基)乙烷、2,6- 双(2-羟基-5-甲基苄基)-4- 甲基苯酚和 4-(4-[1,1- 双(4-羟基苯基)乙基]苯)- α , α - 二甲基苄基苯酚、四(4-羟基苯基)- 甲烷、双(2,4- 二羟基苯基)酮、1,4- 双(4,4- 二羟基三苯基甲基)苯；偏苯三酸；均苯四酸；二苯甲酮四甲酸及其酰氯 (acid chlorite)。在使用三官能或更高官能芳族化合物的情况下，优选使用 1,1,1- 三(4-羟基苯基)乙烷和 1,1,1- 三(3,5- 二甲基-4-羟基苯基)乙烷。

[0048] 聚碳酸酯或共聚碳酸酯的特征是用 Ubbelohde 毛细管粘度计 (毛细管类型 0C) 测量，在二氯甲烷中的聚合物溶液中测定的相对溶液粘度。聚合物浓度为 5g/1，测量在 25°C 温度进行。优选的相对溶液粘度应为 1.190 至 1.230 范围。

[0049] 一元醇或多元醇的高级脂肪酸酯可作为释放剂加到本发明的热塑性树脂中。通过掺合一元醇或多元醇的高级脂肪酸酯，可改进在模塑热塑性树脂时从模的释放能力，并且在模塑光盘基片中的释放负荷变小，从而能够防止由释放失败导致的光盘基片变形和间隔错位。热塑性树脂的熔体流动性也得到改进。

[0050] 释放剂的有效量优选小于 2000ppm 树脂重量，更优选小于 1000ppm 树脂重量，尤其优选小于 500ppm。

[0051] 优选的高级脂肪酸酯为甘油单硬脂酸酯和甘油单棕榈酸酯及其混合物。

[0052] 也可向热塑性树脂加入基于磷的热稳定剂。适合的基于磷的热稳定剂例如亚磷酸、磷酸、亚膦酸、膦酸及其酯。具体地讲，可指出亚磷酸酯化合物，例如亚磷酸三苯酯、亚磷酸三(壬基苯基)酯、亚磷酸三(2,4- 二叔丁基苯基)酯、亚磷酸三癸酯、亚磷酸三辛酯、亚磷酸三(十八烷基)酯、亚磷酸二癸基单苯酯、亚磷酸二辛基单苯酯、亚磷酸二异丙基单苯酯、亚磷酸单丁基二苯酯、亚磷酸单癸基二苯酯、亚磷酸单辛基二苯酯、二亚磷酸双(2,6- 二-叔丁基-4- 甲基苯基)季戊四醇酯、亚磷酸 2,2- 亚甲基双(4,6- 二-叔丁基苯基)辛酯、二亚磷酸双(壬基苯基)季戊四醇酯和二亚磷酸双(2,4- 二-叔丁基苯基)季戊四醇酯；及磷酸酯化合物，例如磷酸三丁酯、磷酸三甲酯、磷酸三(甲苯基)酯、磷酸三苯酯、磷酸三(氯苯基)酯、磷酸三乙酯、磷酸二苯基甲苯酯、磷酸二苯基单正联苯酯、磷酸三丁氧基乙酯、磷酸二丁酯和磷酸二异丙酯。另外的基于磷的热稳定剂例如四(2,4- 二-叔丁基苯基)-4,4'- 亚联苯基二亚膦酸酯、四(2,4- 二-叔丁基苯基)-3,3'- 亚联苯基二亚膦酸酯和双(2,4- 二叔丁基苯基)-4- 亚联苯基亚膦酸酯。其中亚膦酸三(壬基苯基)酯、二亚磷酸二硬脂基季戊四醇酯、二亚磷酸双(2,4- 二-叔丁基苯基)季戊四醇酯、亚磷酸三(2,4- 二-叔丁基苯基)酯、磷酸三苯酯、磷酸三甲酯、四(2,4- 二-叔丁基苯基)-4,4'- 亚联苯基二亚膦酸酯和双(2,4- 二叔丁基苯基)-4- 亚联苯基亚膦酸酯是优选的。这些热稳定剂可单独或作为混合物使用。

[0053] 如果加入,这些热稳定剂的量优选对 100 重量份热塑性树脂组合物为 0.0001 至 0.05 重量份,更优选 0.0005 至 0.02 重量份,最优选 0.001 至 0.01 重量份。

[0054] 可向本发明的热塑性树脂中加入一般已知的抗氧化剂,以防止氧化。抗氧化剂的实例为基于苯酚的抗氧化剂。抗氧化剂的量优选为基于热塑性树脂的 0.0001 至 0.05 重量 %。

[0055] 可将上述添加剂和其它添加剂,例如光学稳定剂、着色剂、抗静电剂和润滑剂加入本发明的光盘基片所用的树脂中,其限度使得由各个聚碳酸酯树脂组合物制成的光盘满足高旋转速度试验的要求。

[0056] 也可使用聚碳酸酯与其它热塑性树脂的掺合物,只要由各个掺合物制成的光盘满足高旋转速度试验的要求。其它热塑性树脂可选自丙烯酸类树脂,优选聚甲基丙烯酸甲酯;聚苯乙烯树脂,作为均聚物或作为与选自异戊二烯和丁二烯或其混合物的其它单体的共聚物或嵌段共聚物;或聚环烯烃树脂,优选烯烃的聚合产物,例如具有降冰片烯结构的部分。

[0057] 在本发明树脂组合物的制备中,可想到在溶液或熔体中进行聚碳酸酯树脂的混合和/或聚碳酸酯树脂和其它树脂的混合。至于混合的装置,在聚合物溶液阶段,主要使用装配有搅拌器的容器,对于第二种情况,可使用例如转鼓、双圆筒混合机、Nauter 混合机、班伯里密炼机、捏和辊或挤出机的设备。无论哪种情况,可使用任何技术,这些技术不受特别限制。

[0058] 制备 DVD 基片的方法

为了从用于光学记录介质的上述树脂制造基片,用装配有压模的注模机(包括注射压缩模塑机)通过注射模塑形成光盘基片,所述压模具有满足光学记录介质和表面精密度所需规格的间隔和凹槽。光盘基片的厚度一般为 0.55 至 0.60mm。此注模机可以为一般使用的机器,但为了防止产生碳化物并改进光盘基片的可靠性,优选为由其圆筒和螺杆具有低树脂附着力、耐腐蚀性和抗磨性的材料制成的机器。考虑到本发明的目的,模塑步骤的环境优选尽可能洁净。要模塑的材料应完全干燥以去除水,并且应避免可能导致熔融树脂分解的停留也很重要。

[0059] 以下描述制造本发明的光学记录介质的标准光盘基片,所述方法用于试验部分,且不限制本发明的范围。使用装配有塑模和压模的注模机,由聚碳酸酯注模具有 120mm 直径和 0.60mm 厚度的光盘基片。

[0060] 制备染料和反射层的方法

DVD 可记录光盘通过旋涂方法用有机染料作为记录层。对于此方法,通过在有机溶剂中增溶染料粉末来制备新鲜的染料溶液。在模塑过程后,将所有基片转移到温度受控输送机,以达到基片双侧的有效冷却,准备好进行进一步染料涂布过程。在线(inline)染料涂布装置通常装配有数个自动化染料投料站,用于在各个开槽的基片上涂覆精确控制的染料层。旋涂过程应严格在气候受控的环境下。为了从凝聚的染料膜去除残余溶剂,将染料涂布的基片进料至具有红外灯的干燥烘箱。

[0061] 为了在染料涂布的基片上沉积高反射性金属层,在干燥过程后使用高速溅射模块。最常用的材料为银,或银合金。最强有力和优化的喷镀金属器可在整个目标寿命中提供均匀的溅射。通过在金属化后向边缘投料清洁溶剂,去除染料涂布和金属化的层 0 基片的边缘上的残余染料。

[0062] 粘合方法描述

在线方法

将金属化和边缘清洁的层 0 基片转移到粘合装置,其中粘合剂材料通过旋涂粘合方法在光盘上投料。随后,在上面放置另一个空白层 1 基片。利用优化旋转方法,使粘合剂在两个基片之间分配。选择粘合剂的剂量,以便使通过此方法得到的粘合剂层厚度在 30 和 60 μm 之间,优选在 40 和 50 μm 之间。

[0063] 使光盘曝露于紫外辐射,以通过自由基聚合反应使粘合材料固化。

[0064] 实验

试验程序

粘合剂的粘度

在高速混合机 (Hauschild DAC 150 FVZ) 中制备粘合剂制剂。粘度在 25 $^{\circ}\text{C}$ 在振动流变仪 (Firma Anton Paar Physica Modell MCR 301) 中测量,振动流变仪装配有设定为 25 $^{\circ}\text{C}$ 的烘箱 CTD 450。使用锥-板几何结构。锥的直径为 50mm,锥角为 1 $^{\circ}$ 。粘度以旋转模式在 10 1/s 的固定剪切速率下测量。

[0065] 测量杨氏模量 E 和品质因子 Q

为了测定在约 10Hz 至 10KHz 频率范围材料的杨氏模量 E 和阻尼相关因子 Q (品质因子),使用类似于 ASTM E 756-05 所述的装置 (图 1)。该测定原理是基于评价关注材料的振动梁的共振频率 f_n 。为了激发共振频率,将梁在一侧夹到压电振动器,它由 1mHz 延伸到 10KHz 范围的模拟白噪声信号驱动。记录梁对该激励的响应,例如:在未夹到压电振动器的梁的末端用激光-多普勒-振动计 (LDV) 测量的速度-频率归一化到例如夹到压电振动器的梁的末端的激励的速度-频率。由此响应曲线,如下计算 E 和 Q:

$$E = \frac{12\rho l^3 f_n^2}{H^3 C_n^2} \quad (3)$$

$$Q = \frac{2f_n}{\Delta f_n} \quad (4)$$

其中:

E= 梁材料的杨氏模量, Pa,

f_n = 模式 n 的共振频率, Hz,

Δf_n = 模式 n 的全带宽半功率 (FWHM), Hz,

C_n = 不夹 (均匀) 梁的模式 n 的系数,

H= 梁在振动方向的厚度, 米,

l= 梁的长度, 米,

n= 模式数: 1, 2, 3, ...,

Q= 梁材料的品质因子, 无量纲,

ρ = 梁的质量密度, kg/m^3 。

[0066] 从根据上述程序制造或者可注射模塑的光盘切出梁。选择梁的宽度 W 为 0.013m,

并选择长度 l , 以使共振频率良好地适合 10Hz 至 10kHz 的测量频率范围。在低于 2000Hz 的测量 f_n 和在高于 2000Hz 的测量 f_{n+1} , 用从公式 (3) 和公式 (4) 提取的各个值通过线性内插计算在 2000Hz 的 E 和 Q 的值。

[0067] 质量密度

利用 Mettler 密度试剂盒 Mettler AT 250 H66765, 用乙醇作为浸没液体, 在室温对光盘切出的样品测量质量密度 ρ 。或者, 从光盘切出已知宽度 W 、已知高 H 和已知长度 l 的光盘的矩形片。通过用天平 (例如, Mettler AT 250) 称重测定光盘的矩形片的质量 m , 并通过 $\rho = m / (l \cdot H \cdot W)$ 计算 ρ 。

[0068] 测量径向和切向偏差

径向偏差度量光盘表面与基准平面的角偏差。其定义为在垂直于基准平面的入射光和反射光之间沿径向测量的角。用来自 Basler 的离线光盘扫描仪 (Modell: Basler Reference 100) 测量光盘的径向偏差。取对光盘测量的最大标称值。

[0069] DVD+R(SL) 光盘的 TE 和 FE 信号评价

用 DVD 写入器 (Plextor®-PX755A 1.01) 评价 TE/FE 性能。用随写入器提供的 PlexTools Professional V2.32a 软件进行评价。用另一种 DVD 写入器 (BenQ® 1650 BCIB) 评价 TE/FE 性能。用随写入器提供的 QScan 软件进行评价。测量必须对未记录的光盘以 16X 速度进行。对各光盘扫描取 TE 和 FE 的最大测量值。

[0070] TE/FE 试验为对于空白 DVD 可记录光盘的写试验 (以模拟模式), 测量光盘的机械特征。FE (聚焦误差) 表示拾取器能够多好地在光盘表面上聚焦激光束。TE (跟踪误差) 表示拾取器能够多好地跟踪光盘的螺旋轨迹。如果数值太高, 就有发生写误差的机会。在此情况下, 应以较低速度进行记录。此试验将以模拟模式在最高记录速度进行。在试验结束, 试验结果将指示此光盘是否可在最高速度以良好的品质写入。

[0071] 粘合剂 A) 的描述

粘合剂 AA) 为氨基甲酸乙酯丙烯酸酯 Desmolux® XP2513, 购自 Bayer Material Science AG, Leverkusen, DE。

[0072] 粘合剂 AH) 为 Ebecryl® 600, 购自 Cytec Surface Specialities SA/NV; Ebecryl® 600 为标准双酚 A 环氧丙烯酸酯。

[0073] 其它粘合剂如下描绘合成。对于这些合成, 除非另外指明, 所有百分数均以重量计。

[0074] 根据 DIN EN ISO 11909, 通过在与丁胺反应后用 0.1mol/l 盐酸回滴, 进行 NCO 含量测定, 用 % 表示。

[0075] 根据 ISO/DIS 3219:1990, 利用购自 Haake, DE 的板-板旋转粘度计 RotoVisko 1, 进行粘合剂的粘度测量。在进行实验时普遍的 23°C 环境温度, 称为 RT。

[0076] 制备氨基甲酸乙酯丙烯酸酯粘合剂 AD

向具有回流冷凝器、可加热油浴、机械搅拌器、气通 (air traversal) (2l/h)、内部温度计和滴液漏斗的 2000ml 四颈玻璃烧瓶加入 275.1g 双-(4-异氰酸酯基环己烯) 甲烷 (Desmodur® W, Bayer Material Science AG, Leverkusen, DE)。加入 0.50g 二月桂酸二丁基锡 (Desmorapid® Z, Bayer Material Science AG, Leverkusen, DE) 和 1.01g 2,6-二-叔丁基甲基苯酚, 并将混合物加热到 60°C。随后, 以使温度不超过 70°C 的速率, 滴加 722.4g

Tone M100[®] (Dow, Schwalbach, DE)。继续搅拌,直到 NCO 含量下降到低于 0.1%。这得到具有 0.08% NCO 含量和 15400mPas (23°C) 粘度的无色树脂。

[0077] 制备氨基甲酸乙酯丙烯酸酯粘合剂 AE

向具有回流冷凝器、可加热油浴、机械搅拌器、气通 (21/h)、内部温度计和滴液漏斗的 2000ml 四颈玻璃烧瓶加入 366.7g HDI 缩二脲 (Desmodur[®] N3200, Bayer Material Science AG, Leverkusen, DE)。加入 0.49g 二月桂酸二丁基锡 (Desmorapid[®] Z, Bayer Material Science AG, Leverkusen, DE) 和 1.01g 2,6-二-叔丁基甲基苯酚,并将混合物加热到 60°C。随后,以使温度不超过 70°C 的速率,滴加 653.6g Tone M100[®] (Dow, Schwalbach, DE)。继续搅拌,直到 NCO 含量下降到低于 0.15%。这得到具有 0.12% NCO 含量和 38500mPas (23°C) 粘度的无色树脂。

[0078] 制备氨基甲酸乙酯丙烯酸酯粘合剂 AF

向具有回流冷凝器、可加热油浴、机械搅拌器、气通 (21/h)、内部温度计和滴液漏斗的 2000ml 四颈玻璃烧瓶加入 366.7g HDI 缩脲二酮 (Desmodur[®] N3400, Bayer Material Science AG, Leverkusen, DE)。加入 0.50g 二月桂酸二丁基锡 (Desmorapid[®] Z, Bayer Material Science AG, Leverkusen, DE) 和 1.0g 2,6-二-叔丁基甲基苯酚,并将混合物加热到 60°C。随后,以使温度不超过 70°C 的速率,滴加 639.84g Tone M100[®] (Dow, Schwalbach, DE)。继续搅拌,直到 NCO 含量下降到低于 0.1%。这得到具有 0.08% NCO 含量和 9050mPas (23°C) 粘度的无色树脂。

[0079] 制备氨基甲酸乙酯丙烯酸酯粘合剂 AG

向具有回流冷凝器、可加热油浴、机械搅拌器、气通 (21/h)、内部温度计和滴液漏斗的 2000ml 四颈玻璃烧瓶加入 153.22g 六亚甲基二异氰酸酯 (Desmodur[®] H, Bayer Material Science AG, Leverkusen, DE)。加入 0.50g 二月桂酸二丁基锡 (Desmorapid[®] Z, Bayer Material Science AG, Leverkusen, DE) 和 1.0g 2,6-二-叔丁基甲基苯酚,并将混合物加热到 60°C。随后,以使温度不超过 70°C 的速率,滴加 532.95g Oxyester T1136[®] (Degussa AG, Marl, DE),然后滴加 313.73g Tone M100[®] (Dow, Schwalbach, DE)。继续搅拌,直到 NCO 含量下降到低于 0.1%。这得到具有 0% NCO 含量和大于 500000mPas (23°C) 粘度的无色树脂。

[0080] 活性稀释剂 B)

活性稀释剂 BA 为二丙二醇二丙烯酸酯 (比较)。

[0081] 活性稀释剂 BB 为 Laromer[®] POEA, BASF SE, Ludwigshafen, DE (丙烯酸苯氧基乙酯)。

[0082] 活性稀释剂 BC 为 OTA480[®], Cytec Surface Specialities, SA/NV (丙氧基化甘油三丙烯酸酯) (比较)。

[0083] 活性稀释剂 BD 为 Photomer[®] 4039, Cognis, Deutschland GmbH (苯酚-(2,5)乙氧基化丙烯酸酯)。

[0084] 活性稀释剂 BE 为 Photomer[®] 4050, Cognis, Deutschland GmbH (聚乙二醇 200 二丙烯酸酯) (比较)。

[0085] 活性稀释剂 BF 为 Sartomer[®] SR-833 S, Cray Valley, F 的 Sartomer 部门 (三环癸烷二甲醇二丙烯酸酯) (比较)。

[0086] 活性稀释剂 BG 为己二醇二丙烯酸酯 (比较)。

[0087] 活性稀释剂 BH 为 Sartomer® SR 256, Cray Valley, F 的 Sartomer 部门 (丙烯酸 2-(2-乙氧基乙氧基)乙酯)。

[0088] 活性稀释剂 BI 为 Laromer® TBCH BASF SE, Ludwigshafen, DE (丙烯酸 4-叔丁基环己酯)。

[0089] 光引发剂 C)

光引发剂 CA 为 Darocur® 1173, Ciba Lampertheim, DE (1-苯基-2-羟基-2-甲基-1-丙-1-酮)。

[0090] 制备粘合剂制剂

将表 3 中所述的所有粘合剂材料配制成为在 25°C 的粘度为约 480 至 520mPas 的粘合剂 A) 和活性稀释剂 B) 之间的比率。根据本领域的技术人员已知的程序进行粘合剂组分的混合。

[0091] 基片模塑方法描述

使用 Singulus E Mould DVD-R 注模机, 用购自 Bayer MaterialScience AG 的 Makrolon® OD2015 注射模塑具有 120mm 直径和 0.60mm 厚度的光盘基片 (信息基片和空基片), 所述注模机连接到装配有购自 Axxicon 塑模的 ST 塑模的 Singulus Streamline II DVD-R, 并使用 DVD+R 或 DVD-R 压模。使用以下模塑参数, 见表 2。

[0092] 表 2: 根据光盘模塑过程的模塑参数

$T_{cyl}(^{\circ}C)$	$T_{塑模}(^{\circ}C)$	夹力(到)	保持时间(s)	冷却时间(s)	注射速度(cm^3/s)
360	123	32	0.5	2.3	220

T_{cyl} 为熔体温度, $T_{塑模}$ 为塑模温度, 而左边值表示塑模的镜像侧, 右边值表示塑模的压模侧。

[0093] 染料和反射层制备描述

DVD 可记录光盘通过旋涂方法用有机染料作为记录层。通过在作为有机溶剂的四氟丙醇中增溶干燥粉末来制备新鲜的染料溶液。在模塑过程后, 将所有基片转移到温度受控输送机, 以达到基片两侧的有效冷却, 准备好进行进一步染料涂布过程。在线染料涂布装置装配有数个自动化染料投料站, 用于在各个开槽的基片上涂覆精确控制的染料层。旋涂过程应严格在气候受控的环境下。为了从凝聚的染料膜去除残余溶剂, 将染料涂布的基片投料至具有红外灯的干燥烘箱。

[0094] 为了在染料涂布的基片上沉积高反射性金属层, 在干燥过程后使用高速溅射模块。最常用的材料为银, 或银合金。最强有力和优化的喷镀金属器可在整个目标寿命中提供均匀的溅射。通过在金属化后向边缘投料二丙酮醇溶剂, 去除染料涂布和金属化的层 0 基片的边缘上的残余染料。

[0095] 在此情况下, 使用上述 Singulus Streamline II DVD-R 机的下游处理设备。

[0096] DVD+R(SL) 在线粘合方法描述

使用 Singulus Streamline II DVD-R 机, 用以上基片和表 4 中所述的粘合剂制剂制备单层 DVD+R 光盘。粘合方法如下所述。

[0097] 将金属化和边缘清洁的层 0 基片转移到粘合装置, 其中粘合剂材料通过旋涂粘合方法在光盘上投料。投料量为 0.7g。随后, 在上面放置另一个空白层 1 基片。利用优化旋

转方法,使粘合剂在两个基片之间分配。通过此程序达到的粘合剂层厚度为约 45 μm 。粘合剂层厚度由 Basler Scanner REF 100 测量。

[0098] 使光盘在紫外固化站曝露于紫外辐射,以通过自由基聚合反应使粘合材料固化。UV 灯的基准功率为 1.0kW,UV 灯的最大功率为 3.0KW,暴露时间为 1.0 秒。

[0099] 手工粘合方法描述

如下所述制造 600 μm 聚碳酸酯光盘 / 粘合剂 / 600 μm 聚碳酸酯光盘夹层结构。

[0100] 将 600 μm 厚聚碳酸酯光盘基片放在旋涂器 (Karl Suss Model RC 8) 夹盘上。粘合剂用加压的投料系统 (GLT Model ETD 2000 XL) 以 35mm 光盘半径涂覆。旋转速度为 200RPM,投料时间为 1.75 秒。在投料系统中使用的注射器针直径为 1.35mm。投料压力调节到约 1.8 巴,以便在基片上分散全圆的粘合剂材料。将第二 600 μm 厚基片放在用粘合剂涂布的第一基片上。通过使夹层结构在 1500RPM 旋转 10 秒,使粘合剂在两个基片之间分配。

[0101] 在 UV 曝光设备上对粘合剂进行随后交联。将两个基片和其间的粘合剂的夹层结构放在以约 1m/ 分钟的速度移动通过 UV 光源 (Hg 灯,80W) 的输送带上。将约 5J/cm² 的辐射剂量施加到夹层结构,以使粘合剂材料固化,并形成光盘。

[0102] 通过此程序达到的粘合剂层厚度为约 60 μm 。通过从粘合的光盘的总厚度减去两个基片的厚度,测得粘合剂层厚度。所有厚度用螺旋测微器测量。

[0103] 从这些人工粘合的光盘和制成的 DVD+R(SL) 光盘,切割样品以用激光多普勒振动计检验光盘的振动性质。尤其在所述试验杆几何形状,模态振动发生在 270Hz。对于试验下的样品,从数据提取在 270Hz 的阻尼相关 Q 值。Q 为 $1/(3 \tan(\sigma))$,而 $\tan(\sigma)$ 为机械阻尼。这些测量的结果描绘于以下表 3 中。

[0104] 表 3:粘合剂材料的组成和人工粘合的各个 DVD+R 光盘在 270Hz 的 Q 值

实施例	粘合剂 A	重量 分数	活性 稀释剂 B	重量 分数	引发剂 C	重量 分数	Q@270 Hz
1	AA	0.563	BB	0.408	CA	0.029	84
2	AA	0.485	BD	0.486	CA	0.029	68
3	AA	0.679	BH	0.291	CA	0.030	94
6	AD	0.728	BH	0.243	CA	0.029	66
7	AE	0.631	BH	0.340	CA	0.029	99
8	AF	0.728	BH	0.243	CA	0.029	128
9	AG	0.558	BH	0.413	CA	0.029	127
比较 实施例	粘合剂	重量 分数	活性 稀释剂	重量 分数	引发剂	重量 分数	Q@270 Hz
1	AA	0.561	BA	0.410	CA	0.029	258
2	AA	0.291	BC	0.680	CA	0.029	266
3	AA	0.515	BE	0.456	CA	0.029	196
4	AA	0.243	BF	0.728	CA	0.029	290
5	AA	0.602	BG	0.369	CA	0.029	255
6	DIC® 6830						227
7	Sony® SK 6500						199
16	AA	0.483	BH	0.146	CA	0.029	163
	AH	0.196	BG	0.146			

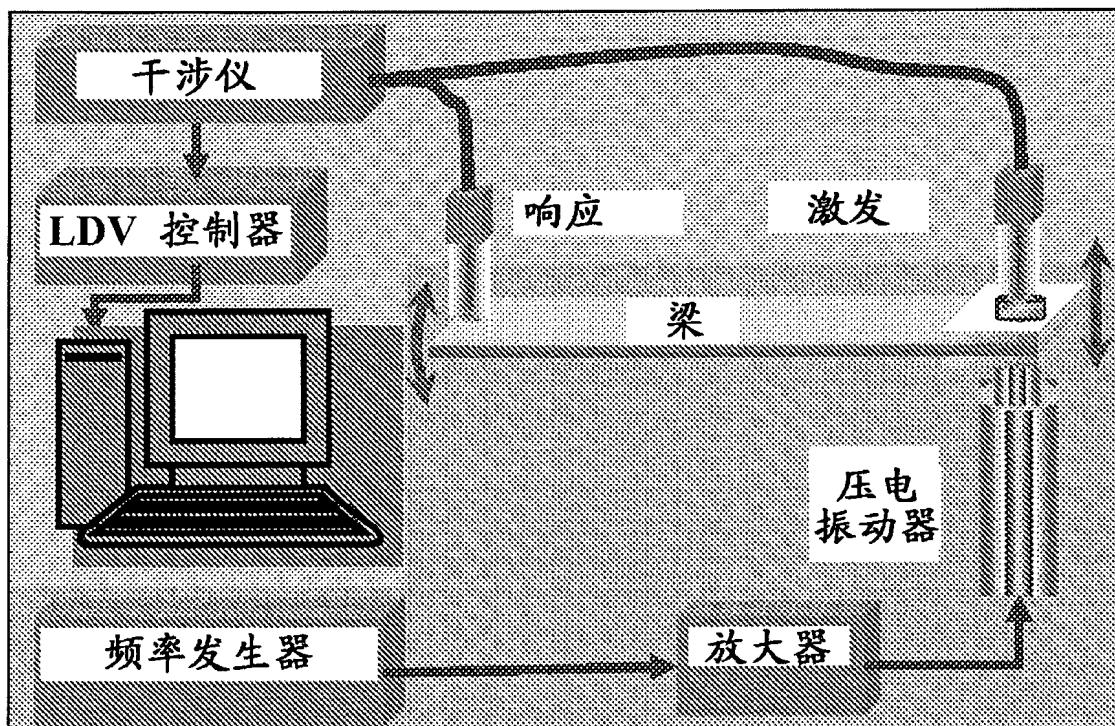
可从表 3 看到,与用实际市售可得的粘合剂材料(如 DIC® 6830 或 Sony® SK 6500(实施例 6-7))和用包含二丙烯酸酯作为活性稀释剂的粘合剂(实施例 1 至 5 和 16)制造的光盘比较,本发明的粘合剂材料改进光盘在 270Hz 的阻尼至少 1.53 的因数。Sony® SK 6500 为特种丙烯酸酯基可旋涂和 UV 可固化 DVD 粘合剂,购自 Sony Chemical & Information Device Corporation Japan,不含本发明的粘合剂。DIC® 6830 为特种丙烯酸酯基可旋涂和 UV 可固化 DVD 粘合剂,购自 DIC Corporation Japan,不含本发明的粘合剂材料。

[0105] 为了证明光盘的较高阻尼与改进的跟踪 (TE) 和聚焦 (FE) 性能的关系,用来自实施例 3 和比较实施例 6 和 7 的粘合剂材料制造 DVD+R(SL) 光盘。如何测量 TE 和 FE 的细节描述于第 9 节,TE 和 FE 信号评价分节。这些试验的结果总结于以下表 4 中。

[0106] 表 4:用专用粘合剂制造的 DVD+R(SL) 在 16X 的 TE/FE 信号

实施例	粘合剂材料	聚碳酸酯基片	最大径向偏差(°)	最大切向偏差(°)	TE	FE	Q@270 Hz
10 Plextools	实施例 3	Makrolon® OD 2015	0.76	0.15	32	16	94
10 QScan	实施例 3	Makrolon® OD 2015	0.76	0.15	251	89	94
比较 实施例	粘合剂材料	聚碳酸酯基片	最大径向偏差(°)	最大切向偏差(°)	TE	FE	Q@270 Hz
17 Plextools	DIC® 6830	Makrolon® OD 2015	0.78	0.22	41	22	227
17 QScan	DIC® 6830	Makrolon® OD 2015	0.78	0.22	291	109	227
18 Plextools	Sony® SK 6500	Makrolon® OD 2015	0.51	0.08	48	20	199

可从以上结果清楚地看到,本发明的粘合剂材料显著改进最终 DVD 光盘的 TE 和 FE 信号。



激光-多普勒-振动计方法的装置

图 1