

DESCRIPTION

LIQUID CRYSTAL RESIN COMPOSITION

Technical Field

[0001]

The present invention relates to a liquid crystal resin composition containing talc.

Background Art

[0002]

For obtaining a liquid crystal resin composition by including talc in a liquid crystal resin, various attempts have been heretofore made. However, resins characterized by high heat resistance and high dimensional accuracy, like liquid crystal resins have had various problems under a high temperature. That is, when a liquid crystal resin composition containing talc in a liquid crystal resin is molded, the liquid crystal resin is heated and melted to mold the liquid crystal resin composition, but the melting temperature of the liquid crystal resin is very high as compared to other resins. A peculiar phenomenon occurs due to such a high temperature. Examples of the peculiar phenomenon include reactions such as thermal decomposition and hydrolysis of a liquid crystal resin, which are caused by the catalytic action of metal impurities in talc, or of

talc itself in some cases. Accordingly, liquid crystal resin compositions containing talc have not had sufficient mechanical characteristics and heat resistance heretofore.

[0003]

Various liquid crystal resin compositions that overcome the above-described problems have been proposed (see, for example, Patent Literatures 1 and 2). Patent Literature 1 describes a heat-resistance resin composition containing 5 to 200 parts by weight of talc, wherein the total content of Fe_2O_3 and Al_2O_3 is less than 1% by weight based on 100 parts by weight of a predetermined copolymerization polyester, and the average particle size is 2.5 to 3.5 μm .

Patent Literature 2 described a composition containing 35% to 65% of an aromatic polyester, and 1% to 60% of talc having only a minimum content of a material that can be decomposed at an elevated temperature.

In both the literatures, however, moldability and heat resistance do not reach a sufficient level. A liquid crystal resin composition having better moldability and heat resistance is desired.

Citation List

Patent Literatures

[0004]

Patent Literature 1: JP 4-202557 A

Patent Literature 2: JP 59-36154 A

Summary of Invention

Technical Problem

[0005]

The present invention has been devised in view of the above-described conventional problems, and an object of the present invention is to provide a liquid crystal resin composition excellent in moldability and heat resistance.

Solution to Problem

[0006]

For achieving the above-described object, the present invention is as follows.

(1) In an aspect, there is provided a liquid crystal resin composition containing 5 to 200 parts by mass of talc based on 100 parts by mass of a liquid crystal resin, wherein the total content of Fe_2O_3 , Al_2O_3 and CaO is 2.5% by mass or less, the total content of Fe_2O_3 and Al_2O_3 is more than 1.0% by mass and not more than 2.0% by mass and the content of CaO is less than 0.5% by mass based on the total solid content of the talc.

[0007]

(2) The liquid crystal resin composition according to (1), wherein the particle size (D50%) of the talc is 4.0 to

20.0 μm as measured by a laser diffraction method.

[0008]

(3) The liquid crystal resin composition according to (1) or (2), wherein the liquid crystal resin is an aromatic polyester or aromatic polyester amide having as a constituent at least one compound selected from the group consisting of an aromatic hydroxycarboxylic acid, an aromatic hydroxyamine and an aromatic diamine.

[0009]

(4) The liquid crystal resin composition according to any one of (1) to (3), wherein the total content of Fe_2O_3 , Al_2O_3 and CaO is not less than 1.0% by mass and not more than 2.0% by mass, the total content of Fe_2O_3 and Al_2O_3 is more than 1.0% by mass and not more than 1.7% by mass and the content of CaO is not less than 0.01% by mass and not more than 0.4% by mass.

Advantageous Effect of Invention

[0010]

According to the present invention, there can be provided a liquid crystal resin composition excellent in moldability and heat resistance.

Description of Embodiments

[0011]

The liquid crystal resin composition of the present invention is a liquid crystal resin composition containing 5 to 200 parts by mass of talc based on 100 parts by mass of a liquid crystal resin, wherein the total content of Fe_2O_3 , Al_2O_3 and CaO is 2.5% by mass or less, the total content of Fe_2O_3 and Al_2O_3 is more than 1.0% by mass and not more than 2.0% by mass and the content of CaO is less than 0.5% by mass based on the total solid content of the talc.

The components will be described in detail below.

[0012]

[Liquid Crystal Resin]

The liquid crystal resin to be used in the present invention refers to a melt-processable polymer having a property that allows an optically anisotropic molten phase to be formed. The property of the anisotropic molten phase can be confirmed by a common polarizing examination method using orthogonal polarizers. More specifically, confirmation of the anisotropic molten phase can be performed in the following manner: a molten sample placed on a Leitz hot stage is observed with a magnification of 40 under a nitrogen atmosphere using a Leitz polarizing microscope. When examined between orthogonal polarizers, the liquid crystal resin applicable to the present invention is usually permeable to polarized light and exhibits optical anisotropy even though the liquid crystal

resin is molten and stationary.

[0013]

The type of the above-mentioned liquid crystal resin is not particularly limited, but an aromatic polyester or an aromatic polyester amide is preferred. A polyester partially containing an aromatic polyester or an aromatic polyester amide in the same molecular chain is similarly preferred. Among them, one having a logarithmic viscosity (I.V.) of preferably about 2.0 dl/g, further preferably 2.0 to 10.0 dl/g when dissolved in pentafluorophenol in a concentration of 0.1% by mass at 60°C is preferably used.

[0014]

The aromatic polyester or aromatic polyester amide as a liquid crystal resin applicable to the present invention is particularly preferably an aromatic polyester or aromatic polyester amide having as a constituent at least one compound selected from the group consisting of an aromatic hydroxycarboxylic acid, an aromatic hydroxyamine and an aromatic diamine.

[0015]

More specifically, the aromatic polyester or aromatic polyester amide is, for example, (1) a polyester composed mainly of one or more of aromatic hydroxycarboxylic acids and derivatives thereof; (2) a polyester composed mainly of (a) one or more of aromatic hydroxycarboxylic acids and

derivatives thereof, (b) one or more of aromatic dicarboxylic acids, cycloaliphatic dicarboxylic acids and derivatives thereof and (c) one or more of aromatic diols, cycloaliphatic diols, aliphatic diols and derivatives thereof; (3) a polyester composed mainly of (a) one or more of aromatic hydroxycarboxylic acids and derivatives thereof, (b) one or more of aromatic hydroxyamines, aromatic diamines and derivatives thereof and (c) one or more of aromatic dicarboxylic acids, cycloaliphatic dicarboxylic acids and derivatives thereof; or (4) a polyester composed mainly of (a) one or more of aromatic hydroxycarboxylic acids and derivatives thereof, (b) one or more of aromatic hydroxyamines, aromatic diamines and derivatives thereof, (c) one or more of aromatic dicarboxylic acids, cycloaliphatic dicarboxylic acids and derivatives thereof and (d) one or more of aromatic diols, cycloaliphatic diols, aliphatic diols and derivatives thereof. Further, a molecular weight regulator may be used in combination of the above-described constituents as necessary.

[0016]

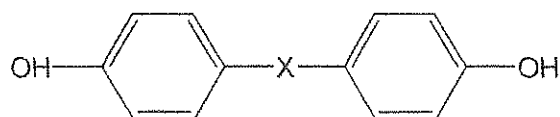
Preferred examples of the specific compound that forms a liquid crystal resin applicable to the present invention is aromatic hydroxycarboxylic acids such as p-hydroxybenzoic acid, 6-hydroxy-2-naphthoic acid; aromatic diols such as 2,6-dihydroxynaphthalene, 1,4-

dihydroxynaphthalene, 4,4'-dihydroxybiphenyl, hydroquinone, resorcin, and compounds represented by the following general formula (I) and the following general formula (II); aromatic dicarboxylic acids such as terephthalic acid, isophthalic acid, 4,4'-diphenyldicarboxylic acid, 2,6-naphthalenedicarboxylic acid, and compounds represented by the following general formula (III); and aromatic amines such as p-aminophenol and p-phenylenediamine.

[0017]

[Chemical Formula 1]

General formula (I)

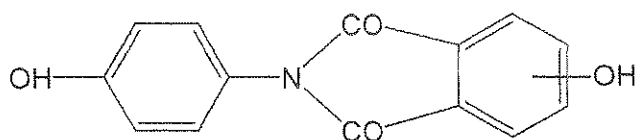


wherein X is a group selected from an alkylene (C1 to C4), an alkylidene, -O-, -SO-, -SO₂-, -S- and -CO-.

[0018]

[Chemical Formula 2]

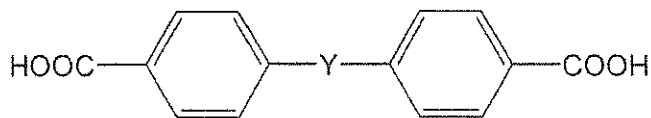
General formula (II)



[0019]

[Chemical Formula 3]

General formula (III)



wherein Y is a group selected from $-(CH_2)_n-$ ($n = 1$ to 4) and $-O(CH_2)_nO-$ ($n = 1$ to 4).

[0020]

The liquid crystal resin to be used in the present invention can be synthesized directly from the above-mentioned monomer compound (or mixture of monomers) by a known method using a polymerization method or an ester-exchange method, but a melt polymerization method or a slurry polymerization method is usually used. The above-mentioned compounds having an ester forming ability may be used for polymerization in their original form, or may be modified from a precursor into a derivative having the ester forming ability in the pre-stage of polymerization. In polymerization of these compounds, various catalysts can be used, and typical examples include dialkyl tin oxides, diaryl tin oxides, titanium dioxide, alkoxy titanium silicic acid salts, titanium alcoholates, alkali and alkali earth metal salts of carboxylic acids, and Lewis acid salts such as BF_3 . The amount of a catalyst used is generally about 0.001 to 1% by mass, particularly preferably about

0.01 to 0.2% by mass based on the total mass of monomers. If necessary, the molecular weight of the polymer produced by the above-mentioned polymerization method can be increased by subjecting the polymer to solid phase polymerization in which heating is performed under a reduced pressure or in an inert gas.

[0021]

The melt viscosity of the liquid crystal resin obtained by the above-mentioned method is not particularly limited. Generally, a liquid crystal resin having a melt viscosity of not less than 10 Pa·s and not more than 600 Pa·s at a shear rate of 1000 sec⁻¹ at a molding temperature can be used. However, a liquid crystal resin having a high viscosity in itself is not preferred because fluidity is extremely deteriorated. The liquid crystal resin may be a mixture of two or more liquid crystal resins.

[0022]

[Talc]

The talc to be used in the present invention is one in which the total content of Fe₂O₃, Al₂O₃ and CaO is 2.5% by mass or less, the total content of Fe₂O₃ and Al₂O₃ is more than 1.0% by mass and not more than 2.0% by mass and the content of CaO is less than 0.5% by mass based on the total solid content of the talc. That is, the talc to be used in the present invention contains, in addition to SiO₂

and MgO as main components thereof, at least one of Fe_2O_3 , Al_2O_3 and CaO, with each of the components being contained with a content falling within the above-described range.

[0023]

When the total content of Fe_2O_3 , Al_2O_3 and CaO is more than 2.5% by mass in the talc, moldability and heat resistance are deteriorated. The total content of Fe_2O_3 , Al_2O_3 and CaO is preferably not less than 1.0% by mass and not more than 2.0% by mass.

It is difficult to obtain talc in which the total content of Fe_2O_3 and Al_2O_3 is 1.0% by mass or less, and when the total content of Fe_2O_3 and Al_2O_3 is more than 2.0% by mass in the talc, moldability and heat resistance are deteriorated. The total content of Fe_2O_3 and Al_2O_3 is preferably more than 1.0% by mass and not more than 1.7% by mass.

When the content of CaO is 0.5% by mass or more, moldability and heat resistance are deteriorated. The content of CaO is preferably not less than 0.01% by mass and not more than 0.4% by mass.

[0024]

In the present invention, the particle size (D50%) measured by a laser diffraction method is preferably 4.0 to 20.0 μm , more preferably 10 to 18 μm for preventing warp deformation and maintaining fluidity.

[0025]

On the other hand, in the present invention, the talc is contained in an amount of 5 to 200 parts by mass based on 100 parts by mass of the liquid crystal resin. When the content of the talc is less than 5 parts by mass, the warp deformation preventing effect is reduced, and when the content of the talc is more than 200 parts by mass, fluidity is deteriorated. The content of the talc is preferably 5 to 100 parts by mass, more preferably 10 to 40 parts by mass.

[0026]

As the talc, commercial products such as Crown Talc PP manufactured by Matsumurasangyo Co., Ltd and MS-K manufactured by Nippon Talc Co., Ltd can be used.

[0027]

[Other Components]

In the present invention, an inorganic filling agent other than talc may be blended. Examples thereof include glass fibers, asbestos fibers, silica fibers, silica/alumina fibers, potassium titanate fibers, boron fibers, silicon carbide fibers, carbon fibers, calcium carbonates such as light calcium carbonate, heavy and pulverized calcium carbonate and special calcium-based filling materials, nepheline and syenite fine powders, calcined clays such as montmorillonite and bentonite, clays

(aluminum silicate powders) such as silane-modified clay, silica (silicon dioxide) powders such as those of fused silica, synthetic silica and crystalline silica, silicic acid-containing compounds such as diatomaceous earth and silica sand, crushed products of minerals such as pumice powders, pumice balloons, slate powders and mica powders, alumina-containing compounds such as alumina, alumina colloids (alumina sols), alumina white and aluminum sulfate, minerals such as barium sulfate, lithopone, calcium sulfate, molybdenum disulfide and graphite (black lead), glass-based fillers such as glass beads, glass hollow bodies, glass flakes and foamed glass beads, fly ash spheres, volcanic glass hollow bodies, synthetic inorganic hollow bodies, single crystal potassium titanate, carbon nanotubes, carbon hollow spheres, carbon 64 fullerene, anthracite powders, artificial ice stone (cryolite), titanium oxide, magnesium oxide, basic magnesium, dolomite, potassium titanate, calcium sulfite, mica, asbestos, calcium silicate, ammonium powders and molybdenum sulfide.

These inorganic filling agents may be used alone, or used in combination of two or more thereof.

[0028]

The method for obtaining a molded article using the liquid crystal resin composition of the present invention is not particularly limited, and a known method can be

employed. For example, a molded article can be obtained in the following manner: the liquid crystal resin composition of the present invention is introduced into an extruder, and melted and kneaded to pelletize the liquid crystal resin composition, and the pellet thus obtained is introduced into an injection molding machine equipped with a predetermined mold, and injection-molded.

The melting temperature in pelletization is preferably 250 to 400°C, more preferably 300 to 380°C.

Examples

[0029]

The present invention will be described further in detail below by way of examples, but the present invention is not limited to the examples below.

[0030]

[Examples 1 to 6 and Comparative examples 1 to 5]

100 parts by mass of a liquid crystal resin (LCP; VECTRA (registered trademark) LCP E950i manufactured by Polyplastics Co., Ltd.), 27 parts by mass of glass fibers (ECS04T-790DE (chopped strand fibers having an average fiber diameter of 6 μm and a length of 3 mm) manufactured by Nippon Electric Glass Company, Limited), 55 parts by mass of talc and 0.55 parts by mass of a mold release agent (pentaerythritol tetrabehenate, Nissan Elector WEP-5 manufactured by NOF CORPORATION) were melted and kneaded

using a twin-screw extruder (TEX-30 α manufactured by The Japan Steel Works, Ltd.), and thereby pelletized. In examples and comparative examples, talcs having compositions described in Table 1 were used. In Table 1, "Fe + Al + Ca" and "Fe + Al" refer to the total content of Fe₂O₃, Al₂O₃ and CaO and the total content of Fe₂O₃ and Al₂O₃, respectively, based on the total solid content of talc. The content of each component contained in talc is a value obtained by performing analysis in accordance with JIS M8851, and the particle size is a value obtained by performing measurement by a laser diffraction method.

[0031]

For the obtained resin composition (pellet), the following evaluation tests were conducted.

(1) Melt Viscosity

The pellet was measured for the apparent melt viscosity in accordance with ISO 11443 at a shear rate of 1000 sec⁻¹ with the cylinder temperature set to a melting point +20°C using a capillary type rheometer (Capirograph 1D manufactured by Toyo Seiki Seisaku-Sho, Ltd.; piston diameter: 10 mm). For the measurement, an orifice having an inner diameter of 1 mm and a length of 20 mm was used. The measurement results are shown in Table 1.

(2) Blister Test

The pellet was molded into a 1/32 combustion test

piece (having a weld) under conditions that ensured a molding cycle of 20 seconds using an injection molding machine (SE 100DU manufactured by Sumitomo Heavy Industries, Ltd.). The obtained test piece was held in a hot press at a specified temperature for 5 minutes, and the surface of the test piece was observed. A maximum temperature that did not cause blistering on the surface was determined as a BFT (blister free temperature). Each of pieces obtained by cutting the test piece at the weld portion was defined as one specimen, the specified temperature was set at an interval of 10°C within a range of 250 to 300°C. The measurement results are shown in Table 1.

(3) Metering Time

The pellet was molded at a screw rotation number of 150 rpm using an injection molding machine (SE 30DUZ manufactured by Sumitomo Heavy Industries, Ltd.; screw diameter ϕ : 18mm; metering stroke length: 55 mm), metering times for 20 shots were measured for each sample, and an average value was obtained. The obtained values are shown in Table 1. A sample having a short metering time is determined to have better moldability.

[0032]

[Table 1]

Talc	Composition (% by mass)	Example 1	Example 2	Example 3	Example 4	Example 5	Example 6	Comparative Example 1	Comparative Example 2	Comparative Example 3	Comparative Example 4	Comparative Example 5	
		SiO ₂	59.51	61.14	60.31	60.17	58.73	60.48	57.87	58.52	58.96	59.24	59.16
		MgO	28.72	28.66	29.19	30.09	28.49	28.82	29.46	28.77	29.75	29.1	29.7
		CaO	0.30	0.07	0.27	0.31	0.14	0.23	1.14	0.87	0.88	0.71	0.51
		Al ₂ O ₃	0.80	0.72	0.65	0.77	0.70	0.88	0.93	0.91	0.94	0.92	1.17
		Fe ₂ O ₃	0.54	0.54	0.53	0.50	0.92	0.53	0.55	0.55	0.55	0.57	0.58
		Ca+Al+Fe	1.64	1.33	1.45	1.58	1.76	1.64	2.62	2.33	2.37	2.20	2.26
	Al+Fe	1.34	1.26	1.18	1.27	1.62	1.41	1.48	1.46	1.49	1.49	1.75	
	Particle size (μm)	12.80	14.92	11.83	12.20	15.62	12.80	10.41	11.62	11.63	10.00	10.76	
	Melt viscosity(Pa·s)	45	46	45	44	46	43	36	35	35	36	36	
	BFT (°C)	280	280	270	270	280	270	260	260	270	260	260	
	Metering time(s)	19.81	19.95	21.14	21.18	19.45	19.39	22.58	24.05	24.26	24.02	23.81	

[0033]

From Table 1, it is apparent that in Examples 1 to 6, excellent heat resistance is obtained because the result of the blister test is 270°C or higher for every example, and excellent moldability is obtained because the metering time is at most slightly longer than 21 seconds. On the other hand, in Comparative Examples 1 to 5, the result of the blister test is as low as 260°C except for Comparative Example 3, and the metering time exceeds 22 seconds for every comparative example, so that good results cannot be obtained for both moldability and heat resistance.

CLAIMS

1. A liquid crystal resin composition containing 5 to 200 parts by mass of talc based on 100 parts by mass of a liquid crystal resin,

wherein the total content of Fe_2O_3 , Al_2O_3 and CaO is 2.5% by mass or less, the total content of Fe_2O_3 and Al_2O_3 is more than 1.0% by mass and not more than 2.0% by mass and the content of CaO is less than 0.5% by mass based on the total solid content of the talc.

2. The liquid crystal resin composition according to claim 1, wherein the particle size (D50%) of the talc is 4.0 to 20.0 μm as measured by a laser diffraction method.