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(56) Related Art
**MONTEDO, O. R. K., et al., "Low thermal expansion sintered LZSA glass-ceramics", American Ceramic Society Bulletin, Vo1. 87., No. 7. (2008), pages 34-40.**
**DE OLIVEIRA, A. P. N., et. al., "Sintering and crystallization of a glass powder in the Li2O-ZrO2-SiO2 system", Journal of the American Ceramic Society. Vol. 81, No. 3 (1998) pages 777-780.**
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**(54) Title**: LITHIUM DISILICATE GLASS CERAMICS, METHOD FOR THE PRODUCTION THEREOF AND USE THEREOF

**(54) Bezeichnung** : LITHIUMDISILIKAT-GLASKERAMIK, VERFAHREN ZU DEREN HERSTELLUNG UND DEREN VERWENDUNG

**(57) Abstract**: The invention relates to glass ceramics on the basis of the lithium disilicate system, which can be easily mechanically machined in an intermediate stage of the crystallization and which represent high-strength, highly translucent and chemically stable glass ceramics after complete crystallization. Furthermore, the invention relates to a method for producing said glass ceramics. The glass ceramics according to the invention are used as dental material.

**(57) Zusammenfassung**: Die Erfindung betrifft Glaskeramiken auf Basis des Lithiumdisilikat-Systems, die in einer Zwischenstufe der Kristallisation einfach mechanisch bearbeitbar sind und nach der vollständigen Kristallisation eine hochfeste, hoch transluzente und chemisch stabile Glaskeramik darstellen. Ebenso betrifft die Erfindung ein Verfahren zur Herstellung dieser Glaskeramiken. Verwendung finden die erfindungsgemäßen Glaskeramiken als Dentalmaterial.

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# Lithium disilicate glass-ceramic, method for production thereof and use thereof

The invention relates to glass-ceramics based on the lithium disilicate system which can be mechanically machined easily in an intermediate step of crystallisation and, after complete crystallisation, represent a very strong, highly-translucent and chemically-stable glass-ceramic. Likewise, the invention relates to a method for the production of these glass-ceramics. The glass-ceramics according to the invention are used as dental material.

Lithium disilicate glass-ceramics are well known from the literature and several patents are based on this glass-ceramic system. Thus, for example, self-glazed lithium disilicate glass-ceramic objects for the production of tableware are described in EP-B-536 479, in EP-B-536 572 lithium disilicate glass-ceramics which can be used, by scattering fine-particle coloured glass on the surface thereof, as lining elements for building purposes.

The main focus of patented lithium disilicate glass-ceramics is on dental applications. This is due to the fact that the crystallisation of lithium disilicate crystals is effected via a phase of lesser strength (lithium metasilicate) and the material system is consequently amenable inter alia to chair-side methods (see S. D. Stookey: "Chemical Machining of Photosensitive Glass", Ind. Eng. Chem., 45, pp. 115 – 118 (1993) and S. D. Stookey: "Photosensitively Opacifiable Glass" US-A-2, 684,911 (1954)). Investigations by Borom, e.g. M. -P Borom, A. M. Turkalo, R. H. Doremus: "Strength and Microstructure in Lithium Disilicate Glass-Ceramics", J. Am. Ceream Soc., 58, No. 9 – 10, pp. 385 – 391 (1975) and M. -P. Borom, A. M. Turkalo, R. H. Doremus: "Verfahren zum Herstellen von Glaskeramiken" (Method for the production of glass-ceramics), DE-A-24 51 121 (1974) show that glass-ceramics which comprise lithium metasilicate as main phase have reduced strength in comparison with glass-ceramics which comprise lithium disilicate as single crystalline phase.

This principle was used in order firstly to produce a glass-ceramic, in a two-step crystallisation process, which can be machined well mechanically, e.g. by means of CAD/CAM methods, and to process this subsequently in a second crystallisation step to form dental glass-ceramic. This method is suitable for being able to use dental restorations according to the so-called chair-side method. In the case of this method, an individually adapted crown/onlay/inlay is milled out of a glass-ceramic block after the first crystallisation step by means of CAD/CAM in the dentist's surgery, this is subjected to the second crystallisation step in a special oven and used directly in the first and only dental appointment for the patient (DE 10 2005 028 637).

In addition, in WO-A-95/32678 and US-A-5,507,981, lithium disilicate glass-ceramics were described, which can be processed to form shaped dental products by means of hot-pressing by using a special compressible crucible. Furthermore, there are known, from DE-C-14

21 886, glass-ceramics based on $SiO_2$ and $Li_2O$ which contain large quantities of physiologically very questionable arsenic oxide. Also in US-A-4,515,634 and in FR-A-2 655 264, lithium disilicate glass-ceramics which are suitable for the production of dental crowns and bridges are disclosed.

All known lithium disilicate glass-ceramics display inadequacies in the processing thereof to shaped products and/or in mechanical or visual properties and/or in chemical stability. In particular when used in the dental field, equally high requirements for all the mentioned properties must be fulfilled.

Starting herefrom, it was the object of the present invention to provide a glass-ceramic which has "improved mechanical and optical properties and also improved chemical stability relative to the (glass-) ceramics known from the state of the art.

This object is achieved by the lithium disilicate glass-ceramic having the features of claim 1 and also by the method for the production of this glass-ceramic having the features of claim 6. In claim 9, uses according to the invention are indicated. Likewise, a shaped dental product having the features of claim 10 is provided. The further dependent claims reveal advantageous developments.

Specifically, in accordance with a first aspect of the invention there is provided a lithium disilicate glass-ceramic having the following composition:

55 to 70% by weight of $SiO_2$,

10 to 15% by weight of $Li_2O$,

10 to 20% by weight of the stabiliser selected from the group consisting of $ZrO_2$, $HfO_2$ or mixtures thereof,

0.1 to 5% by weight of $K_2O$,

0.1 to 5% by weight of $Al_2O_3$,

0 to 10% by weight of additives selected from the group consisting of boron oxide, phosphorus oxide, fluorine, sodium oxide, barium oxide, strontium oxide, magnesium oxide, zinc oxide, calcium

oxide, yttrium oxide, titanium oxide, niobium oxide, tantalum oxide, lanthanum oxide and mixtures thereof and also

0 to 10% by weight of colourants,

the stabilizer being present essentially in the amorphous phase.

Preferably, the colourants are glass-colouring oxides and/or pigments.

Preferably, the glass-colouring oxides are selected from the group of the oxides of iron, titanium, cerium, copper, chromium, cobalt, nickel, manganese, selenium, silver, indium, gold, rare earth metals, in particular neodymium, praseodymium, samarium and europium.

Preferably, the pigments are doped spinels.

Preferably the lithium disilicate glass-ceramic has the following composition:

58 to 64% by weight of $SiO_2$,

11 to 13% by weight of $Li_2O$,

10 to 15% by weight of the stabiliser selected from the group consisting of $ZrO_2$, $HfO_2$ or mixtures thereof,

2 to 5% by weight of $K_2O$,

2 to 5% of $Al_2O_3$,

2 to 5% of $P_2O_5$ and also

0 to 5% by weight of additives selected from the group consisting of boron oxide, phosphorus oxide, fluorine, sodium oxide, barium oxide, strontium oxide, magnesium oxide, zinc oxide, calcium oxide, yttrium oxide, titanium oxide, niobium oxide, tantalum oxide, lanthanum oxide and mixtures thereof and also

0 to 10% by weight of colourants.

In accordance with a second aspect of the present invention, there is provided a method for the production of a lithium disilicate glass-ceramic according to one of the preceding claims, in which:

a) an initial glass is produced which comprises the components of the glass-ceramic,

b) the initial glass is subjected to a first heat treatment in order to produce a glass-ceramic which has lithium metasilicate as main crystal phase,

c) the glass-ceramic of b) is subjected to a second heat treatment in which the lithium metasilicate is converted with $SiO_2$ from the glass phase into lithium disilicate and subsequently lithium disilicate is present as main crystal phase.

Preferably, the first heat treatment is effected at a temperature of 620°C to 800°C over a period of time of 1 to 200 min, in particular of 650°C to 750°C over a period of time of 10 to 60 min.

Preferably, the second heat treatment is effected at a temperature of 800°C to 1,040°C over a period of time of 5 to 200 min, in particular of 800°C to 870°C over a period of time of 5 to 30 min.

Also in accordance with the invention, there is provided the use of a lithium disilicate glass-ceramic as described in accordance with the first aspect of the invention, as dental material or as component of a dental material.

Still further, in accordance with the invention there is provided a shaped dental product comprising a lithium disilicate glass-ceramic as described in accordance with the first aspect of the invention, in the form of an inlay, an onlay, a bridge, a pin construction, a veneer, a (partial) crown.

Within the scope of the present invention, glass compositions have been developed which can be prepared in a two-step production process, are easy to machine after the first crystallisation step, in particular by means of CAD/CAM, and, after a very short second crystallisation step, are both highly-transparent and very strong and have better chemical stabilities than the known lithium disilicate glass-ceramics.

It was shown surprisingly that the addition of ZrO2 to certain glass compositions leads to glass-ceramics which can be machined very readily in an intermediate crystallisation step and, in the end state, have excellent strength values, exceptional translucence and significantly increased chemical stabilities.

It was shown that up to 20% by weight of a stabiliser selected from the group consisting of $ZrO_2$, $HfO_2$ or mixtures hereof can be incorporated in the glass without having a significant influence on the structure. Contrary to all expectations, the stabiliser does not hereby crystallise out as a separate crystal phase but remains in the remaining glass phase. As a result of the high proportion in the amorphous phase, the mechanical and chemical stabilities in this phase are hugely improved, which also leads to improved properties in the end product.

In particular the chemical stability can be improved via the composition of the remaining glass phase since the glass phase has a significantly higher solubility than the lithium disilicate and hence represents the weak point with respect to chemical attack. The extremely high solubility of the stabiliser ($ZrO_2$) in the glass phase is in particular remarkable since e.g. zirconium oxide acts in many silicate glass-ceramics as nucleation agent, i.e. crystallises out as first phase during a temperature treatment, and the actually sought crystal phase is facilitated and is deposited in a fine-crystalline manner on these $ZrO_2$ crystals.

As a result of the high proportions of stabiliser which remain essentially in the amorphous phase, the crystalline proportion is correspondingly restricted. As a result, and due to the low crystallite size of the lithium disilicate crystals, good translucence of the materials is produced after the second crystallisation. The translucence is however also further improved by the refractive index of the glass phase being increased in turn by the stabiliser and, consequently, being adapted to the refractive index of the lithium disilicate. In the case of glass-ceramics in which the refractive index of the amorphous matrix phase corresponds to the refractive index of the crystalline phase/phases, very good translucence properties are found, relatively irrespective of the crystallite size. In the glass-ceramics according to the invention, therefore all three points for the production of an extremely translucent glass-ceramic are fulfilled:

- limited crystal phase proportion,
- small crystals (< 500 nm),
- adapted refractive index of amorphous and crystalline phase.

The high proportion of stabiliser has the effect therefore in the glass-ceramic of

- improved chemical stability,
- higher strength values and
- improved translucence in several respects

to corresponding glass-ceramics without or with only a low $ZrO_2$- or $HfO_2$ proportion.

The glass-ceramics according to the invention can be produced preferably by means of a method, in which

a) an initial glass is produced which comprises the components of the glass-ceramic,

b) the initial glass is subjected to a first heat treatment at a first temperature in order to produce a glass-ceramic which has lithium metasilicate as single or main crystal phase and

c) this glass-ceramic is subjected to a second heat treatment in which the lithium metasilicate is converted with $SiO_2$ from the glass phase into lithium disilicate and subsequently lithium disilicate is present as single or main crystal phase.

The crystallisation to form lithium metasilicate preferably takes place at temperatures between 620°C and 800°C, with times between 1 and 200 minutes, preferably between 650°C and 750°C for 10 to 60 minutes.

The crystallisation to form lithium disilicate preferably takes place at temperatures between 800°C and 1,040°C, with times of 5 to 200 minutes, preferably between 800°C and 870°C for 5 to 30 minutes.

The subject according to the invention is intended to be explained in more detail with reference to the subsequent examples without wishing to restrict said subject to the special embodiments shown here.

## Examples 1 to 6

In examples 1 to 6, compositions of glasses with a high zirconium oxide content are indicated, which are converted by a two-step temperature treatment firstly into readily mechanically machinable lithium metasilicate glass-ceramics and subsequently into highly-translucent, very strong and chemically-stable lithium disilicate glass-ceramics.

The compositions with their components are represented in Table 1.

**Table 1**

| | **B1** | **B2** | **B3** | **B4** | **B5** | **B6** |
|---|---|---|---|---|---|---|
| $SiO_2$ | 66.9 | 65.8 | 65.5 | 63.7 | 63.5 | 63.5 |
| $Li_2O$ | 13.9 | 13.7 | 13.6 | 13.2 | 14.4 | 12.9 |
| $ZrO_2$ | 10.0 | 10.0 | 12.0 | 11.7 | 12.7 | 13.5 |
| $Al_2O_3$ | 3.2 | 3.1 | 3.1 | 3.0 | 3.3 | 3.5 |
| $P_2O_5$ | 3.0 | 3.0 | 3.0 | 2.9 | 3.1 | 3.4 |
| $K_2O$ | 2.9 | 2.9 | 2.9 | 2.8 | 3.0 | 3.2 |
| $CeO_2$ | - | 1.0 | - | 2.0 | - | - |
| $Er_2O_3$ | - | 0.2 | - | 0.3 | - | - |
| $Tb_2O_3$ | - | 0.3 | - | 0.3 | - | - |

The glasses were melted at 1,500°C and poured into metal moulds to form blocks. The blocks were stress-relieved at 560°C in the furnace and cooled slowly. For the different characterisation processes, the glass blocks were divided up and subjected to a first crystallisation treatment. For this purpose, the glasses were aged for 10 to 120 minutes at 600°C to 750°C. As a result, glass-ceramics with strength values of 150 MPa to 220 MPa were produced. Exclusively lithium metasilicate was hereby established as crystal phase. In this state, machining by means of CAD/CAM methods is very readily possible.

With a second short crystallisation at 800°C to 950°C for 3 to 15 minutes, recrystallisation of the lithium metasilicate with amorphous $SiO_2$ from the glass phase takes place to form lithium disilicate and the result is an increase in strength to 300 MPa to 450 MPa. In addition to the lithium disilicate phase, a subsidiary crystal phase with a zirconium oxide content can hereby be produced. In addition, also small residues of lithium metasilicate can be present. The unequivocal main crystal phase is lithium disilicate.

In Table 2, the crystallisation conditions of individual glasses and also the resulting crystal phases and strength values are displayed.

**Table 2**

| Glass | B1 | B2 | B3 | B4 | B5 | B6 |
|---|---|---|---|---|---|---|
| 1. Crystallisation | 650°C<br>20 min | 700°C<br>40 min | 650°C<br>30 min | 700°C<br>20 min | 700°C<br>40 min | 700°C<br>40 min |
| 2. Crystallisation | 850°C<br>10 min | 830°C<br>10 min | 870°C<br>20 min | 850°C<br>8 min | 820°C<br>10 min | 830°C<br>10 min |
| Crystal phases | | | | | | |
| - Main phase<br>(> 80%) | disilicate | disilicate | disilicate | disilicate | disilicate | disilicate |
| - Subsidiary phase<br>(< 20%) | - | - | - | - | metasilicate | metasilicate |
| Translucence | excellent | very good | excellent | very good | excellent | excellent |
| 3-point<br>bending strength | 375 MPa | 413 MPa | 380 MPa | 418 MPa | 356 MPa | 385 MPa |

The CLAIMS defining the invention are as follows:

1. Lithium disilicate glass-ceramic having the following composition:

   55 to 70% by weight of $SiO_2$,
   10 to 15% by weight of $Li_2O$,
   10 to 20% by weight of the stabiliser selected from the group consisting of $ZrO_2$, $HfO_2$ or mixtures thereof,
   0.1 to 5% by weight of $K_2O$,
   0.1 to 5% by weight of $Al_2O_3$,
   0 to 10% by weight of additives selected from the group consisting of boron oxide, phosphorus oxide, fluorine, sodium oxide, barium oxide, strontium oxide, magnesium oxide, zinc oxide, calcium oxide, yttrium oxide, titanium oxide, niobium oxide, tantalum oxide, lanthanum oxide and mixtures thereof and also
   0 to 10% by weight of colourants,

   the stabilizer being present essentially in the amorphous phase.

2. Lithium disilicate glass-ceramic according to claim 1,
   characterised in that the colourants are glass-colouring oxides and/or pigments.

3. Lithium disilicate glass-ceramic according to any one of the preceding claims,
   characterised in that the glass-colouring oxides are selected from the group of the oxides of iron, titanium, cerium, copper, chromium, cobalt, nickel, manganese, selenium, silver, indium, gold, rare earth metals, in particular neodymium, praseodymium, samarium and europium.

4. Lithium disilicate glass-ceramic according to any one of the preceding claims,
characterised in that the pigments are doped spinels.

5. Lithium disilicate glass-ceramic according to any one of the preceding claims having the following composition:

58 to 64% by weight of $SiO_2$,
11 to 13% by weight of $Li_2O$,
10 to 15% by weight of the stabiliser selected from the group consisting of $ZrO_2$, $HfO_2$ or mixtures thereof,
2 to 5% by weight of $K_2O$,
2 to 5% of $Al_2O_3$,
2 to 5% of $P_2O_5$ and also
0 to 5% by weight of additives selected from the group consisting of boron oxide, phosphorus oxide, fluorine, sodium oxide, barium oxide, strontium oxide, magnesium oxide, zinc oxide, calcium oxide, yttrium oxide, titanium oxide, niobium oxide, tantalum oxide, lanthanum oxide and mixtures thereof and also
0 to 10% by weight of colourants.

6. Method for the production of a lithium disilicate glass-ceramic according to one of the preceding claims, in which

a) an initial glass is produced which comprises the components of the glass-ceramic,

b) the initial glass is subjected to a first heat treatment in order to produce a glass-ceramic which has lithium metasilicate as main crystal phase,

c) the glass-ceramic of b) is subjected to a second heat treatment in which the lithium metasilicate is converted with

$SiO_2$ from the glass phase into lithium disilicate and subsequently lithium disilicate is present as main crystal phase.

7. Method according to claim 6, characterised in that the first heat treatment is effected at a temperature of 620°C to 800°C over a period of time of 1 to 200 min.

8. Method according to claim 6, characterised in that the first heat treatment is effected at a temperature of 650°C to 750°C over a period of time of 10 to 60 min.

9. Method according to any one of claims 6 to 8, characterised in that the second heat treatment is effected at a temperature of 800°C to 1,040°C over a period of time of 5 to 200 min.

10. Method according to any one of claims 6 to 8, characterised in that the second heat treatment is effected at a temperature of 800°C to 870°C over a period of time of 5 to 30 min.

11. Use of the lithium disilicate glass-ceramic according to any one of claims 1 to 5 as dental material or as component of a dental material.

12. Shaped dental product comprising a lithium disilicate glass-ceramic according to any one of claims 1 to 5, in particular in the form of an inlay, an onlay, a bridge, a pin construction, a veneer, a (partial) crown.