

TRANSLATION FROM GERMAN

Patent ApplicationDisk-shaped WafersSpecification

The invention relates to disk-shaped wafers based on an inorganic sorbent and a binder, with a thickness of less than 700 microns, which are characterized by high mechanical strength and low brittleness and which are able to effectively sorb inorganic and organic gases or vapors.

The production of wafers, especially tablets, based on zeolites and binders is already known. Thus, according to JP-A-61 15 5216, zeolite tablets are produced by mixing a zeolite, a binder and a lubricant and extruding the mixture. They are obviously tablets with the same dimensions in all directions.

JP-A-56063818 discloses production of zeolite tablets for use as gas adsorbents, powdered zeolite dried at 105 to 110°C being mixed with 8.1% by weight bentonite powder and being kneaded with a 4% aqueous urea solution. The mixture is tableted, dried and calcined at 510°C. The increase in the compressive strength is dictated by the urea content.

JP-A-55 16 5144 discloses kneading zeolite powder for refrigerant aggregates in powder form with bentonite and water, extruding the mixture and forming round particles with a diameter from 0.8 to 10 mm.

According to JP-A-55 10 4913, zeolite in the Na form is mixed with 25% by weight clay, kneaded with water, extruded, calcined at 650°C, immersed in a calcium chloride solution, washed, dried at 110°C and activated at 400°C. The tablets are used as desiccants.

According to JP-A-603 2572, zeolite powder is mixed with kaolin and Na (or NH₄)-hydroxyethyl cellulose, shaped, dried and calcined at 650°C, in order to increase the strength of the zeolite tablets.

According to JP-A-21 44 121, deodorants are extruded by extruding zeolite powders or grains with calcium chloride or bentonite and water, thereupon the mixture is tableted and the tablets calcined.

According to JP-A-63 218 234, desiccants are produced by extruding a mixture of microporous particles (for example, gypsum, cement, ceramic powder) and an inorganic or organic filler, such as CaCl₂, LiCl, bentonite, zeolites, PVA or other water-soluble polymers. The mixture is tableted and then hardened.

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According to JP-A-60 132 643, zeolite tablets are produced as desiccants using 20% sepiolite as the binder. The mixture is kneaded with water, tabletted, dried at 150°C and calcined at 550°C. The tablets have an improved drying effect compared to bentonite tablets.

The tablets produced according to the prior art are unsuited for use under spatially restricted conditions and under mechanical stress since they are too thick and too heavy and in terms of mass and surface have overly low sorption power for toxic gases and vapors. With the processes and mixtures according to the prior art overly brittle wafers are obtained which crumble especially after firing.

It is known that electroluminescent devices work properly over a longer time only when a desiccant is present. This can be attributed to the sensitivity of the electrodes, especially of the cathodes, for example to moisture (the cathodes consist of Ca or Mg alloys). Therefore these devices are sealed as well as possible under a protective gas.

In EP 500 382 A2 the use of a moisture absorber in an electroluminescent device is described. The desiccant in the form of a powder or beads is applied to the black silicone resin coating. According to the preferred embodiment the desiccant is placed in a gas-permeable bag.

US-A-5,882,761 likewise describes the use of a desiccant in an electroluminescent device. BaO is used as the preferred desiccant.

The sorbents known from the aforementioned publications have the disadvantage that they can sorb only water vapor. An attack on the cathode can however be triggered by other gases which in addition to water form when the epoxy resin which is used for sealing sets (ammonia, volatile amines). In addition, the action of oxygen also leads to failure of the luminescent components (oxidation of the cathode).

The object of this invention is to make available disk-shaped wafers based on an inorganic sorbent and an inorganic binder, with a very low thickness (less than 700 microns) which in spite of their low thickness have high strength and thus can be installed especially in electronic components in which only limited space is available and which can be exposed to vibrations (for example, electronic display devices in motor vehicles and mobile telephones).

This object is achieved as claimed in the invention by preparing a disk-shaped wafer based on at least one inorganic sorbent and at least one binder, with a thickness of less than 700 microns, which can be obtained by pressing a mixture of or a mixture containing the inorganic sorbent, ~~roughly~~ 20 to 60% by weight of the binder and ~~roughly~~ 10 to 15% by weight water (relative to the overall mixture) at a pressure of at least 70 MPa; and calcination of the resulting green wafer at temperatures of at least ~~roughly~~ 500°C until the water content is largely removed.

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The wafers as claimed in the invention have high strength, low brittleness, high sorption rate and high sorption capacities at low mass. They exhibit low thermal expansion, no wear and can be easily colored by adding pigments during manufacture.

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In order to obtain green wafers as claimed in the invention of sufficient strength, the adherence to indicated limits for the mixing ratio of the sorbent, binder, and water proportion and the compaction pressure is of extreme importance. In order to study the effect of the mixing ratio of the sorbent and binders on the product properties, it is a good idea to use the weight ratio of the dry sorbent to the dry binder as a parameter to eliminate the different water contents of the various sorbents and binders. Moreover it is advantageous in the computation or establishment of the water content to consider only the relatively loosely bound proportion which can be determined by heating to 160°C, since only the amount of this water influences the behavior of the mixture in the pressing process and the quality of the final product, but not the water which is structurally ~~highly~~ bound by the sorbent or binder.

Strongly

To explain this concept the following examples may be used. As the sorbent zeolite 4A is assumed and as a commercially available powder it has a water content of 20% by weight which can only be completely removed above 500°C. As the binder bentonite is used which as a commercially available powder has a water content of 12% by weight which can be completely removed at 160°C. This is common knowledge to one skilled in the art.

If as claimed in claim 1 for example 70 parts zeolite 4A, 20 parts bentonite and 10 parts water are mixed, the weight ratio of dry sorbent to dry binder is

$$(0.8 * 70) / (0.88 * 20) = 3.2$$

The water content, as can be determined by drying at 160°C, is composed of the water which was added to the mixture (10 parts), and the water which is loosely bound by the bentonite:

$$10 + (0.12 * 20) = 12.4$$

This mixture is accordingly determined by the weight ratio of the dry sorbent to the dry binder of 3.2 and the water content of 12.4% by weight.

If as claimed in claim 1 for example 25 parts zeolite 4A, 60 parts bentonite and 15 parts water are mixed, the weight ratio of dry sorbent to dry binder is

$$(0.8 * 25) / (0.88 * 60) = 0.4$$

The water content as can be determined by drying at 160°C is composed of the water which was added to the mixture (15 parts), and the water which is loosely bound by the bentonite:

$$15 + (0.12 * 60) = 22.2$$

This mixture is accordingly determined by the weight ratio of the dry sorbent to the dry binder of 0.4 and the water content of 22.2% by weight.

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When the wafers are calcined, besides the water which is loosely bound by the binder and the water which may be strongly bound by the sorbent, the water which is structurally bound by the binder can also be released. When using bentonite as the binder at the calcination temperature, the hydroxyl groups which are bound to the octahedrally coordinated magnesium and aluminum ions are split off as water. The total amount of water released during calcination thus depends on the ratio of the sorbent to the binder, on the nature of the sorbent and the binder, and the amount of water added. For example, in a mixture which is formed from zeolite 4A as the sorbent and bentonite as the binder, the mixing ratio of the dry sorbent to the dry binder being 1.3 and the water content being between 8 and 20% by weight, ~~roughly~~ *about* between 20 and 32% by weight is released as water during calcination.

Preferably the wafers are kept at the calcination temperature or another suitable elevated temperature until this amount of water has been released and a constant weight has been achieved. Thus, according to one preferred embodiment of the invention, drying or calcination takes place up to a residual moisture content of < 2% by weight (determined at the calcination temperature).

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According to one preferred embodiment of the invention it thus relates to disk-shaped wafers containing at least one inorganic sorbent and at least one binder, with a thickness of less than 700 microns, in the mixture the weight ratio of the dry sorbent to the dry binder being between 3.2 and 0.4. Preferably the water content of the mixture, determined at 160°C, is between ~~roughly~~ 8 and 22% by weight. The wafers can be obtained using a process in which the mixture containing the inorganic sorbent, binder, water and optionally pressing aids, is pressed at a pressure of at least 70 MPa and the resulting green wafers are calcined at temperatures of at least ~~roughly~~ 500°C. Preferably calcination takes place up to a constant weight or up to a residual moisture content of less than or equal to 2% by weight, determined at the calcination temperature. The preferred mixing ratio of dry sorbent to dry binder is between ~~roughly~~ 2.1 and 0.7. *about*

The wafers as claimed in the invention can be produced in automated processes in large numbers per unit of time. They can be easily handled and can be removed from a storage container for example using a so-called "pick-and-place" machines and can be inserted into an electronic device.

The wafers as claimed in the invention are able to sorb not only water vapor, but also other gases (ammonia, amines, oxygen). Since they have a high sorption capacity, the electronic device into which they are inserted need not be sealed completely airtight.

Preferably the inorganic sorbent constitutes a natural or artificial zeolite. But other sorbents can also be used, such as amorphous silicic acid or aluminum hydroxide and mixtures of inorganic sorbents.

In principle, any binder which seem suitable to one skilled in the art in this area can be used as the binder. Preferably a ~~smectic~~ clay, especially bentonite, is used as the binder.

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Likewise the use of other inorganic binders, for example aluminum oxide hydroxide (pseudoboehmite), is possible. But also carbohydrate-based or protein-based organic binders can be used, for example starch, cellulose derivatives (such as CMC or CEC), casein or also synthetic polymers such as PVA, PVP or polyphenols or tannin-containing binders (quebracho). Mixtures of different binders can also be used.

Surprisingly, adding bentonite to the zeolite does not reduce the sorption capacity of the latter. In fact, a synergistic effect can be established, i.e. the absorption of water vapor by the mixture is reduced far less than could be expected purely according to calculations.

The thickness of the wafer is preferably ^{about} roughly 200 to 400 microns, its binder content is preferably roughly 40 to 50% by weight.

The subject matter of the invention is furthermore a process, especially for producing the above defined wafers, which is characterized in that a mixture containing the inorganic sorbent, ^{about} roughly 20 to 60% by weight of the binder, roughly 10 to 15% by weight of water and 0 to 5% by weight of one or more pressing aids is pressed at a pressure of at least 70 MPa and the resulting green wafer is calcined at temperatures of at least roughly 500°C until the water content is largely removed. If the water content of the sorbent or binder used is already sufficient, it is not necessary to add any additional water.

According to one preferred embodiment of the process, the ratio by weight of the dry sorbent and of the dry binder in the mixture is between roughly 3.2 and 0.4. The water content of the mixture, determined at roughly 160°C, is preferably between roughly 8 and 22% by weight.

Preferred process measures consist in that the mixture is dried preferably to a water content of ^{about} roughly 10 to 25% by weight, especially of roughly 12% by weight, whereupon the dried granulate is crushed preferably to a particle size of somewhat less than 250 microns.

It was found that when the mixture is pressed into a wafer the best results can be achieved when the mixture contains not more than 15% by weight, preferably not more than 8% by weight, especially 0% by weight of particles larger than 250 microns, preferably > 200 microns and especially preferably > 150 microns. Moreover, the proportion of particles < 45 microns should not be more than 50% by weight, preferably not more than 30% by weight, and especially preferably not more than 20% by weight. The wafers produced in this way have especially advantageous physical and chemical properties.

The preferably used ^{about} A-zeolite is available in powder form and has a moisture content of roughly 10 to 22% by weight. The zeolite is mixed with bentonite powder with a moisture content of preferably roughly 10 to 20, especially 12% by weight, and with water and this mixture is then granulated.

The water content of the mixture depends on the properties of the bentonite; so much water should be added to the bentonite/zeolite mixture that the mixture can be granulated.

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Preferably a high-efficiency mixer is used for this purpose. The bentonite preferably has a montmorillonite content of $> 80\%$ by weight.

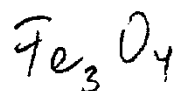
Preferably a fatty-acid salt of a ^dbivalent or trivalent metal, such as calcium or aluminum stearate, is used as the pressing aid.

The preferred pressing pressure is roughly 100 to 1300 MPa.

The wafers are calcined at ^{about} roughly 500 to 900°C, preferably roughly 650°C, until a constant weight is reached.

The wafers can also be calcined under a vacuum, by which permanent gases such as oxygen are also sorbed.

Furthermore, the wafers can contain dyeing pigments, for example Fe_3O_4 .



The subject matter of the invention is furthermore the use of the above defined wafers as inserts into electronic devices or components, such as display devices, especially in electroluminescent components, such as organic light-emitting diodes (LED). But they can also be used in moisture-sensitive liquid crystal displays (LCD).

These devices or components can also be damaged in their operation by inorganic or organic gases or vapors during production or use, and as a result of their design have only very little space available for the sorbent.

These electronic devices or components (for example, display devices in motor vehicles and mobile telephones) are often exposed to strong vibrations, for which reason it is important that the wafers do not break or crumble. Due to their strength it is not necessary to cover the wafers with a gas-permeable film, by which the production of electronic components is simplified.

Compared to BaO, the volume and costs of the electronic component can be distinctly reduced. Thus the wafers, relative to mass, have a higher sorption capacity and sorption rate for water vapor in the required temperature and moisture range within an electronic component. Moreover, when using BaO it must be considered that during the hydration reaction the volume of the material increases by 100%; therefore, within the component additional volume for the expansion of the desiccant should be provided and between the BaO and the electroluminescent layer a film which is permeable to water vapor and which prevents contact between the expanding and possibly crumbling desiccant and the layer should be placed. Conversely, the wafers do not change volume when water vapor is absorbed and they remain mechanically stable, so that making available an additional expansion volume within the component and attaching the protective film can be abandoned.

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In addition, BaO has the disadvantage that it itself and its hydration products react in a highly basic manner; moreover it heats up very dramatically on a local basis when moisture is absorbed and upon direct contact with organic compounds it tends to self-ignite. This limits the choice of polymers for the aforementioned protective film to very expensive ones, for example fluoropolymers, and thus raises the cost of the component. Moreover, when using BaO disposal problems arise, since as a chemical which is harmful to health, it makes disassembly, re-use and disposal of individual parts of the electronic component very difficult.

The wafers as claimed in the invention however can also be used for other purposes, for example as inserts in pharmacy packages, since here only a limited volume is available for accommodating a desiccant. *critical*

The wafers can be present in any shape, for example, round, square, triangular or rectangular or can also contain holes and/or recesses. The wafers as claimed in the invention are free of dust and resistant to wear. They can be produced in ordinary automatic pressing machines in large numbers per unit of time. Presses with multiple tools are used.

The invention is explained by the following examples.

Example 1 (Comparison)

75.2 kg zeolite 4A (water content 20%), 23.8 kg bentonite (water content 12%) and 1 kg calcium stearate are mixed in an high-efficiency mixer for 2 minutes. Then water is added until the viscosity rises dramatically, and mixing continues for another 4 minutes. The mixture is dried at 110°C to a water content of 12% and then granulated (Stokes granulator) and screened (250 microns). 0.22 g of the material with a particle size < 250 microns are pressed with a pressure of 69 MPa into a round wafer. The green wafers are calcined at 650°C for three hours, cooled with the exclusion of moisture, and packaged airtight. The thickness of the wafer decreases by roughly 15 to 25% during calcination.

Product properties:

Thickness: 300 +/- 50 microns

Moisture content (after calcination): < 1%

Scrap in production: ⁹> 80%

Drop test*: 100% fracture, wafer crumbles on the edge

*The so-called drop test is used as a measure for compressive strength, 100 calcined wafers (round wafers with a diameter of 27 mm) being allowed to fall from a height of 1 m with the flat side down. The percentage of broken test specimens is ascertained.

Example 2 (Comparison)

57 kg zeolite 4A (water content 20%), 42 kg bentonite (water content 12%) and 1 kg calcium stearate are mixed in an high-efficiency mixer for 2 minutes. Then water is added until the viscosity rises dramatically, and mixing continues for another 4 minutes. The mixture is dried at 110°C to a water content of 12% and then granulated (Stokes granulator) and screened (250 microns). 0.22 g of the material with a particle size < 250 microns are pressed with a pressure of 69 MPa into a wafer. The green wafers are calcined at 650°C for three hours, cooled with the exclusion of moisture, and packaged airtight.

Product properties:

Thickness: 300 +/- 50 microns

Moisture content (after calcination): < 1%

Scrap in production: 75%

Drop test: 80% fracture

Example 3

57 kg zeolite 4A (water content 20%), 42 kg bentonite (water content 12%) and 1 kg calcium stearate are mixed in an high-efficiency mixer for 2 minutes. Then water is added until the viscosity rises dramatically, and mixing continues for another 4 minutes. The mixture is dried at 110°C to a water content of 12% and then granulated (Stokes granulator) and screened on a 250 micron screen. 0.22 g of the material with a particle size < 250 microns are pressed with a pressure of 72 MPa into a wafer. The green wafers are further treated as in Example 2.

Product properties:

Thickness: 300 +/- 50 microns

Moisture content (after calcination): < 1%

Scrap in production: < 50%

Drop test: 60% fracture

Example 4

57 kg zeolite 4A (water content 20%), 42 kg bentonite (water content 12%) and 1 kg calcium stearate are mixed in an high-efficiency mixer for 2 minutes. Then water is added until the viscosity rises dramatically, and mixing continues for another 4 minutes. The mixture is dried

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at 110°C to a water content of 12%, then granulated (Stokes granulator) and screened on a 250 micron screen. 0.22 g of the material with a particle size < 250 microns are pressed with a pressure of 350 MPa into a wafer. The green wafers are calcined at 650°C for three hours, cooled with the exclusion of moisture, and packaged airtight.

Product properties:

Thickness: 300 +/- 50 microns

Moisture content (after calcination): < 1%

Scrap in production: < 25%

Drop test: 15% fracture

Sorption capacity*	after one hour:	5.4% by weight
	after 5 hours:	7.2% by weight
	after 24 hours:	13.0% by weight

*The sorption capacity for water vapor is determined at 25°C in an atmosphere with 10% humidity.

Example 5

The procedure from Example 4 was repeated with the difference that calcination of the green wafers took place in a vacuum. The calcined wafers had essentially the same product properties as the wafers from Example 4, but in addition exhibited an absorption capacity for oxygen of roughly 5 ml/g (determined in a dry oxygen atmosphere).

Example 6

56.9 kg zeolite 4A (water content 20%), 41.5 kg bentonite (water content 12%), 1 kg calcium stearate and 1 kg quebracho are mixed in an high-efficiency mixer for 2 minutes. Then water is added until the viscosity rises dramatically, and mixing continues for another 4 minutes. The mixture is dried at 110°C to a water content of 12% and then granulated (Stokes granulator) and screened (250 microns). 0.22 g of the material with a particle size < 250 microns are pressed with a pressure of 200 MPa into a wafer. The green wafers are calcined at 650°C for three hours, cooled with the exclusion of moisture, and packaged airtight.

Product properties:

Thickness: 300 +/- 50 microns

Moisture content (after calcination): < 1%

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Scrap in production: < 35%

Drop test: 10% fracture

Example 7

The procedure from Example 4 was repeated with the difference that the pressure used in pressing is 1200 MPa. The drop test yielded 10% fracture. Scrap was < 10%.

Example 8

The procedure from Example 4 was repeated with the difference that 64 kg zeolite 4A, 40 kg bentonite, 5 kg Fe_3O_4 and 1 kg calcium stearate were used. The resulting wafers were dark colored and were used in a LED display as a contrast surface.

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Example 9

57 kg zeolite 4A (water content 20%), 42 kg of a 50/50 mixture of attapulgite and kaolin (water content 12%) and 1 kg calcium stearate are mixed in an high-efficiency mixer for 2 minutes. Then water is added until the viscosity rises dramatically, and mixing continues for another 4 minutes. The mixture is dried to a water content of 12%, then granulated and screened on a 150 micron screen. 0.17 g of the material with a particle size < 150 microns are pressed with a pressure of 200 MPa into a wafer. The green wafers are calcined at 650°C for three hours, cooled with the exclusion of moisture, and packaged airtight.

Product properties:

Thickness: 300 +/- 50 microns

Moisture content (after calcination): < 1%

Scrap in production: 25%

Drop test: 70% fracture

Example 10

An organic electroluminescent component 1 (square, surface 12.9 cm²), as shown in Figure 1, is produced using a wafer (round, diameter 27 mm) from Example 4. After the wafer 2 is mounted on the back wall 3 of the component, it is attached using an adhesive 4 to the glass substrate 5 of the component and sealed as much as possible using the adhesive. Then a microscopic photograph (magnification 50x) of the light emitting part 6 (consisting of the anode

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7, the light emitting layer 8 and the cathode 9) of the component is taken. This photograph does not show any dark (non-luminous) specks which would indicate an attack on the cathode 9.

The component is exposed to a temperature of 85°C and a relative humidity of 85% for 500 h. Then a microscopic photograph of the light emitting part 6 of the component 1 is taken again. Comparison of the two photographs shows that no dark specks formed which would indicate an attack on the cathode 9.

Example 11 (Comparison)

An organic electroluminescent component 1 as in Example 9 is produced using BaO. The covering for the BaO is a water-permeable teflon film which is attached to the rear wall 3 of the component using thin, two-sided adhesive tape. The amount of BaO is set such that the total mass of the BaO, the teflon film and the double-sided adhesive tape corresponds exactly to that of the wafer used in Example 9. Then, as described in Example 9, magnified photographs of the light emitting part before and after storage for 500 h at 85°C and 85% humidity are taken. Comparison of the two photographs shows the distinctly recognizable growth of dark points which indicate an attack on the cathode 9.

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WO 01/44107 A2
Süd-Chemie

CLAIMS

1. Disk-shaped pressed bodies (wafers) based on at least one inorganic sorbent and at least one binder, with a thickness of less than 700 μm , which is obtainable by pressing a mixture containing the inorganic sorbent, about 20 to 60% by weight of the binder and about 10 to 15% by weight water (relative to the overall mixture) and a pressing aid, at a pressure of at least 70 MPa; and calcination of the resulting green wafer at temperatures of at least about 500°C until the water content is largely removed.
2. Wafer as claimed in claim 1, characterized in that the weight ration of the dry sorbent and the dry binder in the mixture is between about 3.2 and 0.2.
3. Wafer as claimed in claims 1 or 2, characterized in that the calcination is done up to a constant weight or up to a residual moisture content of < 2% by weight, determined at the calcination temperature.

4. Wafer as claimed in any one of claims 1 to 3, characterized in that the inorganic sorbent constitutes a natural or artificial zeolite.
5. Wafer as claimed in any one of claims 1 to 4, characterized in that the binder represents a smectitic clay, preferably bentonite.
6. Wafer as claimed in any one of claims 1 to 5, characterized in that the thickness of the wafer is about 200 to 400 μm .
7. Wafer as claimed in any one of claims 1 to 6, characterized in that its binder content is about 40 to 50% by weight.
8. Wafer as claimed in any one of claims 1 to 7, characterized in that the pressure is about 100 to 1300 MPa.
9. Wafer as claimed in any one of claims 1 to 8, characterized in that it has been pressed with a fatty acid salt of a divalent or trivalent metal, as a pressing aid.
10. Wafer as claimed in any one of claims 1 to 9, characterized in that it has been pressed with a tannin-containing binder, preferably quebracho.
11. Wafer as claimed in any one of claims 1 to 10, characterized in that it has been calcined under vacuum.

12. Wafer as claimed in any one of claims 1 to 11, characterized in that it contains a dyeing pigment.
13. Wafer with the solid components as claimed in any one of claims 1 to 12, wherein the water content of the mixture, determined at 160°C, is preferably between about 12.4 and about 22% by weight, but generally between about 8 and 22% by weight.
14. Process for producing disk-shaped pressed bodies (wafers), having a thickness of less than 700 μm , characterized in that a mixture containing at least one inorganic sorbent, about 20 to 60% by weight of at least one binder, about 10 to 15% by weight water, and a pressing aid, is pressed at a temperature of at least about 70 MPa, and the resulting green wafer is calcined at temperatures of at least about 500°C until the water content is largely removed.
15. Process as claimed in claim 14, characterized in that the weight ratio of the dry sorbent and the dry binder in the mixture is between about 3.2 and 0.4.
16. Process as claimed in claims 14 or 15, characterized in that the calcination is done up to a constant weight or up to a residual moisture content of < 2%, determined at the calcination temperature.
17. Process as claimed in any one of claims 14 to 16, characterized in that one or more of the components indicated in claims 2 to 13 are used under the conditions given there.

18. Process as claimed in any one of claims 14 to 17, wherein the water content of the mixture, determined at 160°C, is preferably between about 12.4 and about 22% by weight, but generally between about 8 and 22% by weight.
19. Use of the wafer as claimed in any one of claims 1 to 13, as an insert into electronic devices such as display devices, especially in electroluminescent components.