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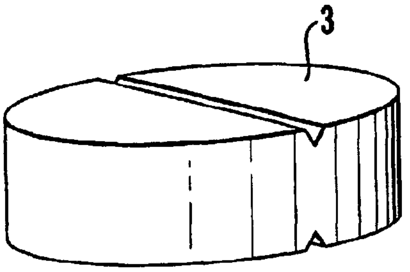

(57) **Abstract:** Raw compact (3) comprising a binding phase of one or more powdered binding agents, which raw compact has the capacity, following saturation with a liquid reacting with binding agents, to form a chemically bound ceramic material, and which raw compact preferably has a degree of compactness of 55-67 percent by volume solid phase. According to the invention, the raw compact (3) also comprises one or more expansion-compensating additives adapted to give the material dimensionally stable long-term properties. The invention also relates to a method of manufacture of the ceramic material, and a compaction device and a compaction body for compacting the saturated raw compacts.

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CHEMICALLY BOUND CERAMIC PRODUCT, METHOD FOR ITS PRODUCTION, TOOL TO BE USED IN EXECUTION OF THE METHOD AND INTERCHANGEABLE PART ON THE TOOL

TECHNICAL FIELD

The present application relates to a raw compact comprising a binding phase of one or more powdered binding agents, which raw compact has the capacity, following saturation with a liquid reacting with the binding agents, to form a chemically bound ceramic material, and which raw compact preferably has a degree of compactness of 55-67 percent by volume solid phase. The primary application of the ceramic material is as dental filling material. The invention also relates to a method for producing said ceramic material, and a tool for handling the raw compact and an interchangeable part for the tool.

BACKGROUND TO THE INVENTION

The present invention relates to binding agent systems of the cement system type, in particular the system CaO-Al_2O_3-(SiO_2)-H_2O. This system is used in the construction industry for exceptionally hard and tough environments, i.e. acid environments with high mechanical stress (R J Mangabhai, Calcium Aluminate Cements, Conference volume, E & F N Spon, London, 1990). By applying rupture mechanical attack methods and advanced powder technology to the system, the generally good profile of features of the base system can be improved considerably. Studies carried out according to the invention and previous works (SE 463 493 and 502 987) have produced a result that indicates great potential for the system for strong and acid-resistant materials such as dental filling materials. No dental filling material existing today meets all the requirements as regards biocompatibility, aesthetics and function that may be set by patients and dental service staff. The situation for various dental filling materials can be summarized as follows: Amalgam has generally good durability, but has shortcomings as far as biocompatibility and aesthetics are concerned. Plastic composites have good workability, but shortcomings with regard to erosion and corrosion and handling for staff (allergy problems have arisen). Plastic composites shrink on hardening, which gives rise to the risk of cracks forming and the onset of decay in the long run. Glass ionomers have a good bond with dentine and enamel, but shortcomings with regard to corrosion and strength. Silicate cement has good compressive strength and aesthetics, but suffers from corrosion and strength problems.

Various types of inlay have good mechanical properties, but are labour-intensive and require gluing.

Below is a description of the requirements which should generally be set for a new, practical dental filling material; good handling ability with easy applicability in a cavity, moulding which permits good modellability, hardening/solidification which is sufficiently fast for filling work and serviceable directly following the visit to the dentist. Furthermore, high strength and corrosion resistance exceeding that of existing filling materials are required, good biocompatibility, good aesthetics and safe handling for staff without allergy-inducing or toxic additives in the materials. In addition, good long-term characteristics with regard to dimensional stability are wanted. This is a problem in particular if the material expands over time, which can cause disastrous tooth breakages as a result.

In SE 463 493 it has been described how a chemically bound ceramic material, for e.g. dental purposes, can be caused to have enhanced strength characteristics in that a powder body consisting of one or more hydraulic binding agents and possible ballast material is compacted at such a high external pressure and so low a temperature that a closely connected raw compact is obtained without sintering reactions on compaction . In this raw compact, the filling density has increased to at least 1.3 times the initial filling density, which is defined as the filling density which is achieved by shaking, vibration and/or light packing of the loose powder in a container. The user of the material prepares the same by saturating the raw compact with a hydrating liquid prior to application of the material or in situ in a cavity, e.g. a tooth cavity.

The material produced according to SE 463 493 has indeed proved to satisfy most requirements that can be set according to the above for dental filling material. However, it has been found that problems can occur with dimensional changes, especially long-term expansion, which according to the above can have disastrous consequences in connection with dental fillings. There is room for further development of the material and its manufacture with regard to other aspects also. Such aspects are for example the degree of compactness of the raw compact, which influences the durability of the raw compact in various directions, workability on application, and the strength of the finished product. Other aspects concern optimization of the raw compact's dimensions and shape and how it is moistened in connection with application. It has been found that problems can occur when the material is hydrated too quickly. There is not then sufficient time, after the raw compact has been mixed with hydration liquid, to mould the material and/or cut into the same, before it solidifies. This problem is accentuated with any accelerator in the hydration liquid. However, the use of accelerator is desirable in itself, since it means that the product quickly becomes polishable, and since the patient can leave the clinic quickly and eat etc.

One aspect that is related to the use of a raw compact for dental fillings is the question of how the raw compact is to be handled purely physically. An instrument is required for gripping the raw compact and transporting it to the cavity that is intended to be filled. A compaction device is likewise required for compaction of the raw compact provided with liquid in the cavity.

Following SE 463 493, it has been shown according to Swedish patent 502 987, that for cement systems complete hydration (which would then lessen the risk of dimensional changes) can take place if complete soaking and subsequent compacting of the cement system take place using a specially designed stopper. However, the method does not prevent dimensional changes which take place afterwards and which are related to phase transformations of hydrate or reactions with the surrounding atmosphere (for example, exhalation air with a higher carbon dioxide content), or other reactions. These reactions and related dimensional changes become more obvious in cases where a high degree of compaction is used in the production of the material. However, a higher degree of packing is normally sought, as this generally gives greater strength. The method according to SE 502 987 may also require a good deal of training before an individual dentist uses it to perfection, which may cause the individual dentist to hesitate to use the technique.

In Yan et al, Characteristics of shrinkage compensation expansive cement containing prehydrated high alumina cement-based expansive additive, Cement and Concrete Research, Vol 24, p 267-276 (1990), the use of calcium aluminate's tendency to expand is described. This article and related works on expansive cements describe the possibilities of making standard cement expand or shrink less using calcium aluminates amongst other things, but do not touch on the problems of long-term expansion of highly compacted cement systems and control of the expansion of calcium aluminates at very low levels, which is a prerequisite for the use of these binding agent systems in applications according to the present invention.

Other immediate works and patents which do not, however, concern the principal field of the present invention are for example SE-B-381 808, EP-A-0 024 056 and EP-A-0 115 058, DE 5 624 489 and US-A-4 689 080.

DISCLOSURE OF THE INVENTION

One object of the present invention is to provide a raw compact of the type stated in the preamble, which raw compact following hydration gives a chemically bound ceramic material that has dimensionally stable long-term properties. The raw compact must also satisfy the requirements stated above for workability and durability, and be easy to handle in connection with its moistening and application in a cavity, e.g. a tooth cavity. The ceramic material formed should also, for dental applications, satisfy the demands that are made on such material according to the above.

This is achieved according to the invention in that the raw compact comprises one or more expansion-compensating additives adapted to give the material dimensionally stable long-term properties.

COMPOSITION OF THE POWDER MATERIAL INCLUDING ADDITIVES

Apart from good mechanical properties, chemical attributes are important for dental applications. In a significant aspect of the invention, calcium aluminates, i.e. double oxides of CaO (calcium oxide) and Al_2O_3 (aluminium oxide) – here and below termed the CA system, which reacts with water, forming calcium aluminate hydrates - are used as the main binding phase. This hydration reaction constitutes the actual setting and hardening process. Conventionally, some type of aggregate (filler particle) is added to the calcium aluminate cement, principally for economic reasons. According to the invention, the choice of the CA cement system, combined with another cement system or phase which interacts with the aluminate cement, or combined with the addition of porous aggregates or soft materials, produces a dimensional change which is less than approx. 0.20 % linearly, often less than 0.10 %. In special cases, the dimensional change may be close to zero expansion.

According to a first embodiment of the invention, the CA system can be used as the only main binding phase or with the addition of another cement binding phase in amounts of less than 30 percent by volume. Admixtures of ordinary Portland cement (OPC cement) or fine-grained silicon dioxide are used advantageously. Since the calcium aluminate cement has a tendency to expand more strongly on harder packing, combinations of CA cement and another phase of said type, with a tendency to shrink, can produce reduced dimensional changes. The CA cement should be present in dental applications as the main phase in the binding phase, as the CA phase contributes to high strength and acid resistance.

It has proved to be the case that the theories regarding reasons for dimensional changes which were put forward in connection with Swedish patent 502 987, i.e. incomplete hydration, do not appear to give a full explanation of the reasons behind the problems with regard to dimensional stability. The background to the present invention is rather the idea that the dimensional changes are linked to phase transformations of hydrate. The statement, which is not to be seen as restrictive for the invention, means that calcium aluminate, when it begins to dissolve on the addition of water, forms a gel which then crystallizes and forms hydrate phases. Due to continued hydration reactions and hydrate transformations, various pure Ca aluminate hydrates such as 10-phase, 8-phase, other less defined hydrate phases or transition phases, and finally 6-phase (katoite) can be present, and in the case of additives containing silicon, Ca-Si aluminate hydrate. 10-phase, 8-phase and 6-phase refer to calcium aluminate phases with 10, 8 or 6 water of crystallization per unit of formula. Phase transformation of the hydrates can lead to dimensional changes, especially expansion, which has been shown by long-term evaluation of cement materials. It has turned out surprisingly to be the case in connection with the present invention that with the addition of a secondary phase containing silicon, preferably ordinary so-called Portland cement (OPC cement with Ca-silicates as main phases) and/or fine crystalline silicon dioxide (which constitutes said first, preferred embodiment of the invention), undesirable phase transformations or changed phase transformation sequences can be avoided in the main, and as a direct consequence of this dimensional changes can be minimized, especially long-term expansion. How the complicated hydration reactions come about in detail is not entirely explained. With the addition of material containing Si, the hydration reactions are modified, leading to dimensionally stable materials.

Surprisingly, it has been found that the positive effects just mentioned on the addition of a secondary phase have an optimum with relatively low addition quantities. The minimum expansion has been attained in this connection when said secondary phase consists of OPC cement and/or fine crystalline silicon dioxide and/or another phase containing Si, preferably in a total content of 1-20 percent by volume and even more preferredly 1-10 percent by volume in the material. Most preferredly, said secondary phase consists of OPC cement in a quantity of 1-5 percent by volume and/or fine crystalline silicon dioxide in a quantity of 1-5 percent by volume. Reference is also made in this context to the examples in this specification.

It has also turned out surprisingly that conventional filler particles providing hardness, e.g. in the form of hard Al_2O_3 particles, can be avoided entirely in the material, or that their use can be minimized, in that hardness is controlled primarily by the hydrate developed. Hydrate transformations are the primary cause of dimensional changes with time, in particular long-term changes. The expansion-compensating additives according to the invention act here on the cement phase, without the influence of any hardness-providing filler particles which may be present. Being able to avoid or minimize the use of hardness-providing filler particles is also due to the fact that any unreacted cement remaining – which was previously considered serious from the expansion viewpoint – only has a slight effect on the expansion. It has been found in connection with the invention that unreacted cement instead works positively, as an in-situ filler material, which contributes to the desired hardness of the material.

According to one embodiment of the invention, however, the raw compact, and thus the finished ceramic material, can contain ballast material, which does not take part in the chemical reactions between the binding phase and the hydration liquid, but which is present as a solid phase in the finished ceramic product. According to one aspect of the invention, the raw compact can therefore contain up to 50 percent by volume of ballast material. This ballast material can for example be of the type described in SE 463 493 and SE 502 987, i.e. fibres of metal, carbon, glass or organic material etc., or continuous crystals, so-called whiskers, of e.g. SiC, Si_3N_4 and/or Al_2O_3.

According to another embodiment of the invention, due to additions of aggregates (filler particles) of a given geometry/shape, porosity and/or softness, the dimensional stability of binding agent systems of interest can be monitored precisely and justified to desired levels, often to low levels or to no dimensional change at all. The situation for the cement system $CaO-Al_2O_3-(SiO_2)-H_2O$, which can be used to advantage as a base material for dental filling material, is described in greater detail below, but the invention relates generally to ceramic binding agent systems in which dimensional stability is critical.

By selecting aggregates (filler particles) in binding agent systems according to the present invention with a specific geometry and porosity, the binding conditions between the binding phase and aggregates can be influenced positively, like the dimensional stability. Porous aggregates and other expansion- or shrinkage-compensating additives thus contribute to the possibilities of being able to justify dimensional changes to a desired level by acting as "expansion vessels".

The function of porous aggregates according to the present invention is thus, with the retention of a high given content of filler particles, to increase the contact surface with the cement phase and distribute this on a smaller propagation area. The expansion which derives from the cement phase is taken up primarily by the porous filler particle in that the cement is given the opportunity to expand inside this. Porous aggregates can consist advantageously of inert ceramic materials such as aluminium oxide, zirconium oxide, titanium oxide or zinc oxide or another oxide or a combination of oxides. The porosity can be present as open or closed porosity or in a combination. In the normal case, the porous particle or aggregate has an open porosity of 20-60 %, preferably 30-50 %. An aggregate size is chosen which is optimally suited to the rupture strength of the materials, but often it has a diameter of less than 20 μm, preferably 5-15 μm. Small porous aggregates or particles contribute in materials of immediate interest to finer surfaces (lower R_a-values) than solid particles of a corresponding size. The pore openings in the aggregates are suited to the penetration capacity of the binding agents. The pore openings are advantageously less than 5 μm, preferably 0.1-5 μm and even more preferredly 1-3 μm.

Porous aggregates or particles of the above named oxides are produced preferably by sintering of fine-grained powder, but not at temperatures too high for the aggregates or particles to be kept porous. Aluminium oxide, for example, is best sintered at around 1500-1600°C. The sintering process is controlled to the desired diameter, porosity and size of pores. Alternatively, the porous aggregates or particles can be produced by mixing fine-grained oxide powder with an agent, e.g. starch, which is made to evaporate so that pores are formed. The material is freeze-granulated by being sprayed and frozen.

In a special case, to be able to take up inner stresses caused by dimensional changes in the binding phase, aggregates with a very high closed porosity can be used, which break in the event of high inner stress and provide internal expansion space. The content of these highly porous particles is limited to a maximum of 5 percent by volume of the binding agent phases. Highly porous microspheres of glass can be used in this case. The highly porous materials are added to the cement mixture in the final stage of the mixing operation to avoid being ground down. In another special case, a very soft particle is chosen as an extra additive, which particle can take up stresses by having an E-modulus lower than that of the binding phase. Various soft polymers, e.g. plastic balls, or hydrate can be used here. When using plastic balls, which are very small, these may also have holes in the middle for further deformability.

According to an aspect of the invention, it has also been found that the dimensional stability of the material can be increased by causing the constituent components to have a highly fine granularity. This also applies to strength aspects. The theory in that case is that particles that are too large have a tendency to lie constricted in the structure, with different accompanying attributes in different directions. According to this aspect of the invention, a fine-grained, finely divided mixture of binding agent raw materials is therefore used, which gives a fine homogeneous microstructure. Small propagation areas for the constituent phases reduce the inner mechanical stress between the phases, and provide a better opportunity to compensate for the internal expansion which can take place in the event of changes of phases, such as continued reaction with the surroundings or phase transformations. The size which can be permitted depends on the level of strength desired, but the grain size should typically lie with a distribution over 0.5-10 µm. The calcium aluminate is caused by grinding for 24-72 hours in the presence of a non-polar liquid to have a grain size in the main of around 2-8 µm, preferably 3-4 µm or around 3 µm. OPC cement, if this is used, is caused by grinding in a corresponding manner, possibly at the same time, to have a grain size in the main of around 4-8 µm, preferably 5-7 µm or around 6 µm. Once grinding has been completed, the non-polar liquid is evaporated from the powder mixture. Fine-grained silicon dioxide, if such is used, would have an even smaller grain size, preferably in the order of magnitude of less than 100 nm, and even more preferredly around 10-50 nm, e.g. around 15 nm, which type of silicon dioxide can be purchased for example as a commercial product, separated in electrostatic filters in the production of silicon.

COMPACTION

The solid constituent parts according to the above are mixed well, at best in the presence of a non-polar liquid, e.g. petroleum ether, acetone or isopropanol, whereupon this non-polar liquid is evaporated from the mixture. The powder mixture is granulated using traditional methods to increase the flow on pressing. The powder mixture, containing expansion-compensating additives and any ballast material, is then compacted to a closely connected raw compact according to the invention. This compaction takes place at high pressure but a low temperature, preferably room temperature, which means that no sintering reactions take place during compaction. Optimization of the degree of compactness is important, since it influences properties such as the durability of the raw compact and the strength of the ceramic material in a positive direction, at the same time as it influences the workability of the raw compact on application in a cavity in a negative direction.

According to a previously known technique (SE 463 493), the raw compact is compacted by cold isostatic compaction (CIP), a powder body being arranged in an impermeable casing, which is exposed to outer pressure in a volume of liquid surrounding the casing. The pressure is stated to exceed 200 MPa, at best 250 MPa minimum. According to the present invention, however, the compaction procedure can be simplified considerably in that it can be carried out as simple mechanical tablet pressing, the raw compacts being pressed in tablet form, one by one, in a mechanical tablet press of a conventional type. That this is possible is due to the fact that the raw compacts according to the present invention are relatively small, which is discussed further below. The size of the raw compacts being relatively small, only a small pressure drop occurs in connection with compaction, which means that simple tablet pressing or tablet punching is sufficient to achieve the desired degrees of compactness. A desired degree of compactness in this connection is a degree of compactness of 55-67 percent by volume solid phase. The degree of compactness is preferably 57-63 percent by volume solid phase and even more preferredly 58-61 percent by volume solid phase, the optimum degree of compactness depending on the size of the raw compact. It is to be noted that these degrees of compactness can in certain cases refer to weighted mean values for constituent phases. Tablet pressing is best executed at a pressure of 40 – 150 MPa, preferably 70 - 110 MPa depending on the table size. The raw compact obtained has a strength (compressive strength) of 0.3 – 5 MPa, preferably 0.5 – 2 MPa, with good edge strength. However, it is not excluded according to the invention that cold isostatic compaction, as described in SE 463 493, can be used in certain cases.

Thanks to tablet pressing to the stated degrees of compactness, a hardness/strength is obtained in the finished ceramic product which is approx. 30-40 % higher than the strength which can be achieved according to the suspension method as described in SE 502 987. At the same time, good workability of the raw compacts is obtained, which workability is better than that which can be achieved with the cold-isostatically compacted raw compacts according to SE 463 493.

According to one aspect of the invention, the raw compact has a largest dimension of 8 mm maximum and a smallest dimension of 0.3 mm minimum, its diameter or width being 1-8 mm, preferably 2-5 mm, and its height being 0.3-5 mm, preferably 0.5-4 mm. To fill a tooth cavity, a number of raw compacts, e.g. 2-5 compacts, are normally needed here. The shape of the raw compacts can be spherical, cylindrical (generally with broken/ chamfered edges) or any other shape which is suitable for tablet pressing and which at the same time gives good strength, e.g. spherical with a cylindrical middle part or a cylinder with a central cut on the flat sides. A tablet-pressed raw compact with an almost spherical shape is easy to grip with instruments from all directions. High cylindrical raw compacts, which are to be applied (in a package) can easily be gripped using a tool (according to below). Low cylindrical raw compacts with a cut on the top and bottom side can be broken in half and fitted into large approximate fillings. The raw compacts can be produced in a number of standard sizes, a minimum size, e.g. with a height of around 0.5-1.5 mm being able to be used for a topmost layer in the tooth filling. This is then easy to pack to a smooth and fine surface without it spreading material to the sides.

According to another aspect of the invention, the shape of the raw compacts can be adapted to a dental drill, so that they fit perfectly into the cavity to be filled. This is applicable to large fillings above all. The tooth cavity is drilled out here using a dental drill with a shape and diameter that match a suitable size of raw compact. The tooth cavity is pretreated and the moistened raw compact inserted directly into the matching hole in the tooth. The raw compact is compacted and packed further into the cavity. Even large fillings, e.g. when a tooth wall is missing, so-called approximate fillings, can be executed in a corresponding manner, the raw compact also being packed against a matrix strip. The advantage of this method is that the strength can be increased further in that the raw compact remains intact right up to being finally packed against the cavity wall or matrix strip. Material wastage is also reduced. In these cases, it is advantageous to use the upper part in the compaction interval, i.e. 61-67 percent by volume solid phase, more preferredly 63-67 percent by volume solid phase.

According to an alternative embodiment, the possibilities of an individualized filling for each cavity are improved further. Here a negative impression is made of the cavity in a suitable impression material, preferably A-silicone mass, whereupon a positive model of the tooth and cavity is produced. Into this die and also exact model of the tooth cavity a raw compact is compacted, which is then steeped in hydration liquid as below, and then applied directly to the tooth cavity as a completely individualized inlay. Subsequent packing with ensuing break-up of the raw compact is suitably avoided. In these cases, it is advantageous to use the upper part in the compaction interval, i.e. 62-67 percent by volume solid phase, more preferredly 64-67 percent by volume solid phase.

APPLICATION IN A CAVITY

According to one aspect of the invention, the development of strength in the ceramic material can be speeded up, so that the material can be polished and thus finished quickly, the time taken for each patient being shortened and the patient quickly being able to use the repaired tooth, e.g for eating etc. Contradictorily, satisfactory time can be obtained according to the invention simultaneously for shaping and cutting the material before it is hydrated. This is possible on the one hand thanks to the use of an accelerator for hydration of the material, and on the other to the method used for packing the raw compacts into the cavity.

Regarding the use of an accelerator, it has been found that using something of this kind accelerates hydration, and at the same time gives a higher early strength in the material. The hydration process is however affected very little by the accelerator during the first minutes (approx. 2-3 minutes) after the raw compact has been saturated with the hydration liquid, meaning that shaping and cutting of the material can be carried out under not too great a time pressure. The time for shaping is also advantageously extended in that *one* raw compact is immersed at a time, at least partly, in the hydration liquid for at least 5-15 seconds, preferably at least 10 seconds and up to 30 seconds. The liquid is permitted here to be absorbed by capillary forces acting in the raw compact, the raw compact preferably being gripped by means of an insertion instrument in connection with its being immersed in the liquid. The amount of liquid absorbed is automatically at least 90-95 % of that required for complete hydration, meaning that the raw compact takes up 15-22 % of liquid calculated on the amount of powder depending on the degree of compactness. That this is the case is due to the fact that the degree of compactness of the raw compact has been optimized, so that due to capillary forces it absorbs 90-95 % of the required quantity of liquid. Such a quantity of liquid gives good compactability. An improved wetting capacity is obtained preferably by preconditioning of the powder mixture and/or of the raw compact at temperatures exceeding 150°C. Any remaining superficial liquid (visible to the eye) on the raw compact is then dried off, e.g. by bringing the saturated raw compact quite quickly into contact with a moistened serviette. Complete hydration then takes place in the cavity, in connection with saliva secretion during hardening of the material. The raw compact can also be saturated in liquid, dried off against a moistened serviette and then applied to the cavity using an instrument.

According to this preferred embodiment of the invention, a first raw compact is thus caused to be saturated by the liquid, in order then to be packed into a cavity, preferably by means of a filling tool, whereupon a second raw compact is caused to be saturated by the liquid, in order then to be packed into said cavity, preferably by means of the same filling tool. Following this, any further raw compacts are caused to be saturated by the liquid, in order then to be packed into the cavity, preferably by means of the same filling tool, at best until the cavity is filled with moist raw compacts packed together, which are then permitted to harden to produce the chemically bound ceramic material. Thanks to the raw compacts being saturated one at a time with hydration liquid, hydration thus commences sequentially for the individual raw compacts, as these are wetted and packed in the cavity. Compared with the method shown in SE 463 493 and in SE 502 987, in which a whole raw pressed cake is wetted, in order then to be broken into pieces and packed in the cavity, a substantial amount of time is thus gained for the moulding. The last material in the saturated raw pressed cake according to previously known techniques will of course have time to begin to hydrate even before it is packed into the cavity and packed together there, possibly resulting in the worst case in uneven packing of the material in the cavity, with the attendant impairment of quality. According to the present invention, however, the material hydrates as it is packed in the cavity. If an accelerator is used, the hydration reaction is accelerated here also, according to the above, after the initial 2-3 minutes, so that a very strong and fast-hardening ceramic material is obtained. The strength can be developed functionally after just 20-60 minutes.

The accelerator preferably consists of a salt of an alkali metal, which is dissolved in the hydration liquid (normally water). Most preferredly, a salt of lithium is used, e.g. lithium chloride or lithium carbonate. The content can be 0.1-1 g/l, preferably at least 0.2 g/l, even more preferredly at least 0.4 g/l and even more preferredly at least 0.8 g/l of water. The higher the content, the quicker the hydration and the greater the strength obtained in the material. Thanks to the sequential wetting and packing, a high content of accelerator can be used without a shortage of time occurring for the application and packing of the material into the cavity. Alternatively, the accelerator, the salt of alkali metal, can be mixed in solid form into the powder mixture before this is compacted. In this connection, the content of accelerator can consist of 10 to 50 ppm, preferably 20 to 30 ppm in the powder mixture. Since the accelerator is present in this manner in solid form, a further small but perhaps important time gain can be achieved in that the dissolution of the salt following saturation of the raw compact by hydration liquid takes up further time.

When the cavity has been filled with compressed raw compacts, and any top layer formed of one or more thin raw compacts, according to the above, final packing and the removal of any surplus liquid in situ is carried out by means of a compacting tool. Normally there will not be any surplus of hydration liquid, due to the fact that the degree of compactness/ porosity of the raw compact has been optimized so that the quantity of liquid which is automatically absorbed by the capillary forces in the raw compact corresponds to the required quantity of liquid for moulding and initial hydration. However, other liquid may have ended up on the material in connection with its packing into the cavity, e.g. saliva, which is why it is expedient to use a demoistening compaction device/tool for the final compaction. The demoistening operating part of the tool consists here of a hard, porous material, into which any surplus liquid is absorbed at the same time as the tool compresses the raw compact further into the cavity.

Following final packing, subsequent polishing, e.g. grinding of a free surface of the chemically bound ceramic material facing towards the oral cavity can be carried out, preferably within 3-10 minutes, even more preferredly within 3-7 minutes after compaction of the moist raw compacts has been completed.

TOOL FOR INSERTION AND FINAL COMPACTION

The invention also relates to a tool for insertion of a raw compact into a cavity. The term insertion refers to a raw compact being gripped, held, introduced into the cavity that is intended to be filled and left/attached there.

The insertion tool according to the invention comprises a cylindrical sheath and preferably a nozzle or head with an internal diameter adapted according to a diameter of a given raw compact. The sheath has an open first short end for receiving the raw compact, and a piston arranged in the sheath, which piston is displaceable in the axial direction of the sheath, for transferring the raw compact to the cavity.

According to one aspect of the tool, a minimum internal diameter of the cylindrical sheath, or nozzle, is less than the diameter of the given raw compact, the sheath, at its first short end or nozzle, having an internal chamfer of approx. 20°, the largest diameter of which exceeds the diameter of the given raw compact.

According to a preferred embodiment of the tool, it is also adapted, when not gripping a raw compact, to grip instead and hold a compaction body, which has a diameter adapted to the internal diameter of the sheath or nozzle and consists of a relatively hard, porous material, due to which the tool forms a demoistening compaction device for compaction in the cavity of one or more raw compacts saturated with said liquid. Due to this, the same tool can thus be used for insertion of the raw compacts into the cavity, and for the final demoistening packing together (compaction) of the raw compacts in the cavity.

The compaction body consists according to the invention of a material in the group consisting of porous ceramic materials, porous polymer materials, porous metal materials and porous wood materials, preferably a material consisting of a hardwood material. The material for the compaction body should have pores with a smaller diameter than the powder grains in the material that is to be compressed. It has been found surprisingly that beech wood functions excellently as a material for the compaction body. The compaction body at best has a diameter that corresponds to the most commonly used diameter of raw compacts, preferably 1-8 mm, even more preferredly 2-5 mm. By means of the compaction device provided with the compaction body, an advantageously combined pressing and suction effect is obtained on the raw compacts packed into the cavity.

BRIEF DESCRIPTION OF DRAWINGS

Some of the aspects according to the invention will be described further below with reference to the enclosed figures, of which:

Figs. 1a-c	show some conceivable embodiments of the raw compact according to the invention;
Figs. 2a-c	show, from the side or from above, a tooth that is drilled and provided with one or more raw compacts according to the invention;
Figs. 3a-b	show a model/die of a tooth with a cavity, and a raw compact which has been moulded in the model/die;
Fig. 4	shows strength as a function of time for some varying concentrations of accelerator in the hydration liquid;
Fig. 5	shows a view in perspective of a first embodiment of the tool, i.e. the insertion instrument, including raw compact;
Fig. 6	shows a side view of the tool in Fig. 5 in cross-section, including raw compact;
Fig. 7	shows the head of the tool according to Fig. 6, in cross-section;
Fig. 8	shows the tip of the head according to Fig. 7, in section, including piston and raw compact;
Fig. 9	shows a side view of two variants of a head for alternative embodiments of the tool;
Fig. 10	shows a side view of yet another variant of a head for an alternative embodiment of the tool;
Fig. 11	shows a side view of a head for a tool according to the invention, which grips a compacting body; and
Fig. 12	shows expansion as a function of time for a ceramic material produced according to the invention.

DETAILED DESCRIPTION OF THE INVENTION

Figs. 1a-c show some conceivable forms of a raw compact according to the invention, to be precise a spherical shape with a cylindrical middle part, a high cylindrical form or a low cylindrical form with a cut for simplified division if so desired.

Figs. 2a-b show how a tooth 1 is drilled out with a drill 2 of a selected diameter, whereupon a cylindrical raw compact 3 of a corresponding diameter can be inserted into the cavity 4 formed in the tooth 1. Fig. 2b also shows the extent of an original attack of decay 5. Fig. 2c shows how several raw compacts 3 can be inserted into a large cavity 5.

Fig. 3a shows a positive model 6 of a tooth, with a cavity 5. This model 6 has been produced from a negative impression (not shown) of an individual tooth with cavity. In a compaction stage for a raw compact, the model 6 is used as a mould or die, a raw compact 3 (Fig. 3b) being obtained that is perfectly suited to the individual tooth with its cavity. This raw compact 3 is placed following moistening into the cavity in the tooth and then preferably not packed any further.

Fig. 4 shows the hardness achieved (measured as HV 100g) in the ceramic material formed as a function of time with some varying concentrations of accelerator, in this case lithium chloride, in the hydration liquid, which consists in this case of water. As is evident, the hardness is developed more quickly and to higher final values the higher the concentration of accelerator used. The levels low, medium and high refer to 1 10^{-4} g, 2 10^{-4} g and 3 10^{-4} g respectively of lithium salt/g of formed product.

Figs. 5 and 6 show a tool 7, which can have an external design that can be the same as for conventional insertion tools and can be produced in plastic or metal, preferably steel. The tool comprises a cylindrical sheath 8 and a piston 9 arranged in the sheath, which piston is displaceable in the axial direction of the sheath. The piston 9 is enclosed in a coil spring 10, which prevents the piston being led too far through the sheath 8. The tool 7, more precisely the piston 9, is also provided with a pressing surface 11 for pressing by a finger, e.g. the thumb, of the user. A projecting dolly 12 is best arranged on the sheath to be gripped by two other fingers, e.g. the index finger and middle finger. The sheath 8 in the embodiment shown is bent to achieve the best means of access to the oral cavity. The tip

13 of the tool is best formed by a head 14, which can be detachable, preferably threaded, so that the tool can be used for raw compacts of a number of different dimensions.

So that it is not too difficult to grip the raw compact, the sheath 8 has an internal chamfer 15 by the head 14, the greatest diameter D of which chamfer exceeds the diameter of a raw compact 3 of a given size and the minimum diameter d of which is less than the diameter of the given raw compact 3, according to Figs. 7 and 8. The chamfer 15 suitably has an angle of around 20° relative to the centre line of the head 14. Due to this, the raw compact can be introduced easily into the tool, and nevertheless held steadily in place in the same. Fig. 8 shows how the raw compact is held firmly by means of the chamfer 15, it also being indicated how the piston 9 can be displaced so that it pushes the raw compact 3 out of the head 14.

According to Fig. 9, the tool, or more precisely the head 14, can be provided with perforations, e.g. in the form of slots 16 or holes 17, with a view to increasing the uptake of hydration liquid when the gripped raw compact 3 is partly immersed in the liquid. A number of slots 16 or holes 17 are here arranged around the circumference of the head 14. An alternative, according to Fig. 10, is to provide the head 14 with a recess 18 in the wall, which suitably extends a little way around the circumference of the head 14 and is arranged a little way, e.g. 3 mm, up from the short end of the head.

The embodiments according to Fig. 9 and 10 are only conceivable variants. In normal cases no perforation or recess is required, since the capillary action of the raw compact is so good that the necessary quantity of liquid is absorbed even when only the lower, projecting part of the raw compact 3 is immersed in the liquid.

Fig. 11 shows how the tool 7 with the head 14 can also grip a compaction body 19, for final compaction of the raw compacts in the cavity. This compaction body 19 consists according to the previous description of a hard, porous material, and has a diameter that is adapted to be gripped by the tool 7.

EXAMPLE 1

A series of experiments was performed to study the effect on expansion, in particular long-term expansion, of various expansion-compensating additives.

Description of raw materials:

Calcium aluminate of the phases $CaO.Al_2O_3$ and $CaO.2Al_2O_3$ forming part of e.g. Ca-aluminate cement (Alcoa or LaFarge), standard cement (Cementa), fine-grained silicon dioxide (Aldrich) and glass spheres (Sil-cell, Stauss GmbH). Al_2O_3 (Sumitomo, AKP 30), ZrO_2 (3-mol % Y_2O_3) from Toyo Soda.

Porous particles, produced in-house from fine-grained Al oxide (Sumitomo, AKP 30) (aggregate diameter approx. 15 micrometres)

The examples under a) – h) describe

a) calcium aluminate's long-term expansion with completely hydrated aluminate without additives, but with hardness-providing filler particles (reference)
b) effect of fine granularity of raw cement materials
c) effect of secondary phase, OPC cement
d) effect of secondary phase, fine-grained Si oxide
e) effect of porous aggregate on b)
f) effect of porous aggregate on c)
g) effect of a combination of OPC and fine-grained Si oxide
h) effect of a combination of various additives
i) effect of Si-containing secondary phases on a pure cement system without hardness-providing filler particles
j) effect of hardness-providing filler particles on i)

Calcium aluminates, $CaO.Al_2O_3$ and $CaO.2Al_2O_3$, with a molecular ratio of approx. 1:1 are mixed with filler particles and secondary additives (all quantities specified are in relation to the quantity of calcium aluminate) as stated below. When "aluminium oxide" is referred to, without the type of particles being specified, conventional hardness-providing filler particles are meant.

a) Addition of 40 percent by volume aluminium oxide, grinding time 24 h. The cement was ground for 20 h beforehand.
b) Addition of 40 percent by volume aluminium oxide, grinding time 24 h. The cement was ground for 80 h beforehand.

c) Addition of 40 percent by volume aluminium oxide, grinding time 24 h. The cement was ground according to b) above beforehand. 15 percent by volume OPC (ordinary Portland cement/standard cement) was added to the calcium aluminate.

d) Addition of 40 percent by volume aluminium oxide, grinding time 24 h. A secondary phase in the form of 10 percent by volume fine-grained silicon dioxide was added to the ground calcium aluminate according to b) above.

e) Addition of 20 percent by volume aluminium oxide, grinding time 24 h. The cement was ground according to b) above. 20 percent by volume of porous aluminium oxide aggregates(produced in-house) was added only after a grinding time of 20 h.

f) Addition of 20 percent by volume aluminium oxide + 20 percent by volume aluminium oxide as porous particles (aggregates), grinding time 24 h, but the aggregates were only added after 20 h. The cement was ground according to b) above, but with the addition of a secondary phase in the form of 15 percent by volume OPC.

g) Addition of 40 percent by volume aluminium oxide, grinding time 24 h. The cement was ground according to b) above. 5 percent by volume of OPC and 5 percent by volume of fine-grained silicon dioxide were added to the calcium aluminate.

h) Addition of 20 percent by volume aluminium oxide + 20 percent by volume aluminium oxide as porous particles, grinding time 24 h, but the aggregates were only added after 20 h. A secondary phase in the form of 5 percent by volume of OPC and 5 percent by volume of fine-grained silicon dioxide and 0.5 percent by volume of glass spheres was added to the calcium aluminate in this case.

i) Addition of secondary phases in the form of 5 percent by volume of OPC and 5 percent by volume of fine-grained silicon dioxide, grinding time 24 h. The cement was ground beforehand for 80 h.

j) Addition of secondary phases in the form of 5 percent by volume of OPC and 5 percent by volume of fine-grained silicon dioxide and hardness-providing filler particles of ZrO_2 of 10 percent by volume, grinding time 24 h. The cement was ground beforehand for 80 h.

The mixtures were ground in a ball mill with inert grinding balls of silicon nitride with a coefficient of fullness of 35 %. Isopropanol was used as a liquid. Following evaporation of the solvent, materials a) – h) were admixed with water, dewatered and tamped with a stopper into holes with a diameter of 4 mm in a container that permitted measurement of the dimensions in an optical microscope. The materials were kept moist at 37°C between test measurements, which were performed continuously up to 180 days.

The results are reported in the table below.

Sample description	Expansion in % after				
	1d	20d	80d	120d	180d
a	0	0.12	0.68	0.82	0.83
b	0	0.22	0.41	0.48	0.48
c	0	0.11	0.23	0.26	0.26
d	0	0.12	0.13	0.13	0.13
e	0	0.15	0.18	0.21	0.21
f-j	all values under 0.10 %				

Error margin for measurements +-0.02 %.

It is evident from the results that expansion stagnates after approx. 100 days. For the very dimensionally stable materials (expansion under 0.15 % including error margins) no clear point in time for stagnation can be deduced. It is also evident that

- Only an increased grinding time (b) in relation to reference (a) almost halves long-term expansion.
- When a secondary phase in the form of OPC cement in a quantity of 15 percent by volume is also added (c), a further virtual halving of long-term expansion is achieved in relation to (b).
- Long-term expansion is further reduced by a secondary phase in the form of fine-grained silicon dioxide in a quantity of 10 percent by volume (d).
- Improved (reduced) long-term expansion in relation to (b) is also achieved using porous aggregates of aluminium oxide in a quantity of 20 percent by volume (e).
- Extremely low expansion was achieved when using porous particles and a secondary phase of OPC cement in combination.
- Extremely low expansion was achieved when using a secondary phase of both OPC cement and fine-grained silicon dioxide, in combination.
- Extremely low expansion was achieved using porous particles, a secondary phase of both OPC cement and fine-grained silicon dioxide and glass spheres in combination.

- Extremely low expansion was achieved for the pure cement system without hardness-providing filler particles when using only a low content of Si-containing additives.
- Extremely low expansion was achieved for the Ca-aluminate system with low quantities (10 percent by volume) of hardness-providing filler particles, using only Si-containing additives for expansion compensation.

The materials which have extremely low expansion (<0.10 %) have, apart from the expansion properties, a general feature profile which matches the corresponding cement system without additives. These materials have a compressive strength of 170-200 MPa, hardness of H (Vickers 100g) = 110-130 and an extremely high acid resistance.

EXAMPLE 2

Description of raw materials:

Calcium aluminate of the phases $CaO.Al_2O_3$ and $CaO.2Al_2O_3$ forming part of e.g. Ca-aluminate cement (Alcoa or LaFarge), standard cement (Cementa), silicon dioxide (Aldrich) and an inert oxide, ZrO_2 (Aldrich).

Mixing:

The cement phases were ground together for approx. 80 h, whereupon the oxides were added in a final mixing operation of approx. 22 h. Finally, the grinding liquid, consisting of 2-propanol, was evaporated.

Pressing:

The powder mixture was pressed into small tablets at a pressure of approx. 90 MPa in a Korsch tablet punching machine. Tablet diameter 3.00 mm and height 3.00 mm with approx. 20° chamfer 0.35 mm in from the flat sides.

Conditioning:

The tablets were heat-treated at 400°C for 4 h.

Insertion and compaction in the test chamber:

Water with alkali salt was added to the tablets one at a time. The tablets were transferred to the cavity in a special holder and compacted with an ordinary stopper initially, to be tamped finally with a porous compacting device (according to Fig. 11). They were then kept in the test bodies at 37°C in a humid environment.

Result:

Fig. 12 shows the resulting expansion as a function of time for the ceramic material formed. It is confirmed that the expansion values achieved are very low and that expansion levels out after 40 days.

The invention is not restricted to the embodiments described, but can be varied within the scope of the claims. Thus it is understood for example that the invention can also be used in connection with non-dental applications, i.e. in connection with the production of chemically bound ceramic materials for other purposes. A conceivable application area is e.g. a carrier material (substrate) for electronic circuits and a substrate in micromechanics. The product can consist here for example of a thin plate with a diameter of around 2-7 cm, for use as a carrier material for electronic circuits, as a carrier material in micromechanical applications, as a carrier material for biosensors or as a carrier material for optical fibres for producing circuit boards, biosensors or the like. It is also conceivable that the product does not need to include expansion-compensating additives in applications of this kind, which however falls outside the scope of the present invention.

Throughout this specification and the claims which follow, unless the context requires otherwise, the word "comprise", and variations such as "comprises" and "comprising", will be understood to imply the inclusion of a stated integer or step or group of integers or steps but not the exclusion of any other integer or step or group of integers or steps.

The reference to any prior art in this specification is not, and should not be taken as, an acknowledgment or any form of suggestion that that prior art forms part of the common general knowledge in Australia.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. Raw compact comprising a binding phase of one or more powdered binding agents, which raw compact has the capacity following saturation with a liquid reacting with the binding agents to form a chemically bound ceramic material, wherein the raw compact also comprises one or more expansion-compensating additives adapted to give the material dimensionally stable long-term properties and wherein the raw compact has a degree of compactness of 55-67 percent by volume solid phase.

2. Raw compact according to claim 1, wherein the raw compact is substantially free of hardness-providing filler particles.

3. Raw compact according to claim 1, wherein the raw compact comprises up to 50 percent by volume ballast material.

4. Raw compact according to any one of the preceding claims, wherein said binding phase consists at least substantially of calcium aluminate cement.

5. Raw compact according to any one of the preceding claims, wherein said additives consist of one or more additives in the group which consists of porous particles or porous aggregates, soft particles which have an E-modulus which is lower than the E-modulus of the binding phase, and a secondary phase, which secondary phase reacts with the binding phase.

6. Raw compact according to claim 5, wherein said additives consist at least substantially of a secondary phase, said secondary phase consisting of OPC cement and/or fine crystalline silicon dioxide and/or another Si-containing phase in a total quantity of 1-20 percent by volume in the raw compact.

7. Raw compact according to claim 6, wherein said secondary phase consists of OPC cement in a quantity of 1-5 percent by volume and/or fine crystalline silicon dioxide in a quantity of 1-5 percent by volume.

8. Raw compact according to claim 5, wherein said additives consist at least substantially of porous particles or porous aggregates, which consist at least substantially of oxides of Al, Zr, Ti, Si, or Zn and Sn, having a diameter of 2-30 μm, an open porosity of 20-60%, and the pore openings in the particles/aggregates being less than 5 μm.

9. Raw compact according to claim 5, wherein said additives at least substantially consist of porous particles, which porous particles consist at least substantially of microspheres with a high closed porosity, which microspheres are of glass and have a porosity which exceeds 50%, and are present in quantities of less than 2 percent by volume of the raw compact.

10. Raw compact according to any one of the preceding claims, wherein said raw compact has a degree of compactness of 57-63 percent by volume solid phase.

11. Raw compact according to any one of the preceding claims, wherein said raw compact also comprises an accelerator for accelerated reaction between said binding agents and the liquid.

12. Raw compact according to claim 11, wherein said accelerator consists of a salt of an alkali metal, in a quantity of 10 to 50 ppm.

13. Raw compact according to any one of the preceding claims, wherein said binding agents substantially have a grain size of 2-8 μm.

14. Raw compact according to any one of the preceding claims, wherein said raw compact has a largest dimension of 8 mm maximum and a smallest dimension of 0.3mm minimum, its diameter or width being 1-8 mm, and its height being 0.3-5 mm.

15. Raw compact according to any one of the preceding claims, which is intended to be used for dental purposes.

16. Raw compact according to claim 15, wherein said raw compact has dimensions adapted to a dental drill.

17. Raw compact according to any one of claims 1-13, which is intended to be used as a carrier material for electronic circuits, as a carrier material in micromechanical applications, as a carrier material for biosensors or as a carrier material for optical fibres.

18. Raw compact according to any one of the preceding claims, wherein said raw compact has dimensions adapted to an individual cavity into which the raw compact is to be introduced.

19. Method for producing a chemically bound ceramic material by means of reaction between a binding phase of one or more powdered binding agents and a liquid reacting with these binding agents, a powder body comprising said binding agents being compacted into a raw compact in that the powder body, before the liquid is made to saturate the same, is exposed to such a high external pressure and at so low a temperature that a closely connected raw compact is obtained without sintering reactions on compaction wherein, the powder body also comprises one or more expansion-compensating additives adapted to give the material dimensionally stable long-term properties, and wherein the raw compact has a degree of compactness of 55-67 percent by volume solid phase.

20. Method according to claim 19, wherein said binding agents consist at least substantially of calcium aluminate cement, said additives consisting of one or more additives in the group which consists of porous particles or porous aggregates, soft particles which have an E-modulus which is lower than the E-modulus of the binding phase, and a secondary phase, which secondary phase reacts with the binding phase.

21. Method according to claim 19 or 20, wherein said raw compact is caused to have a

degree of compactness of 57-63 percent by volume solid phase.

22. Method according to any one of claims 19-21, wherein the method comprises a tablet pressing stage for each raw compact that is produced, tablet pressing being executed at a pressure of 40-150 MPa.

23. Method according to any one of claims 19-22, wherein said raw compact is formed in that said compaction is executed in a model of a cavity into which the raw compact is to be introduced.

24. Method according to any one of claims 19-23, wherein the powder mixture and/or said raw compact is preconditioned at temperatures exceeding 150°C.

25. Method according to any one of claims 19-24, wherein said liquid is caused to saturate the raw compact in that the raw compact is immersed at least partly in the liquid, preferably for at least 5-15 seconds, preferably at least 10 seconds and up to 30 seconds, so that the liquid is permitted to be absorbed by the capillary forces acting in the raw compact, and any remaining superficial liquid on the raw compact then being dried off.

26. Method according to any one of claims 19-25, wherein said liquid consists of a hydration liquid.

27. Method according to claim 26, wherein the hydration liquid contains an accelerator for the reaction between the binding phase and the liquid, said accelerator consisting of a salt of an alkali metal, in a quantity of 0.1-1 g/l of liquid.

28. Method according to claim 25, wherein a first raw compact is caused to be saturated by the liquid, in order then to be packed into a cavity, by means of a filling tool, whereupon a second raw compact is caused to be saturated by the liquid, in order then to be packed into said cavity, by means of said filling tool, whereupon any further raw

compacts are caused to be saturated by the liquid, in order then to be packed into said cavity, by means of said filling tool until the cavity has been filled with moist, compressed raw compacts, which are then permitted to harden to said chemically bound ceramic material.

29. Method according to claim 28, wherein final packing and the removal of any surplus of liquid is executed in situ by means of a compaction device a part of which acting against the moist, compacted raw compacts consists of a hard, porous material, into which any surplus of liquid is absorbed, at the same time as the moist raw compacts packed together are compacted further in the cavity.

30. Method according to claim 28 or 29, wherein subsequent polishing of a free surface of the chemically bound ceramic material formed is executed within 3-10 minutes after said compaction of the moist raw compacts has been completed.

31. Raw compact according to claim 1 and substantially as hereinbefore described with reference to the accompanying drawings and/or Examples.

32. Method according to claim 19 and substantially as hereinbefore described with reference to the accompanying drawings and/or Examples.

DATED: 27 September, 2005

Doxa Aktiebolag

by DAVIES COLLISON CAVE
Patent Attorneys for the Applicant(s)

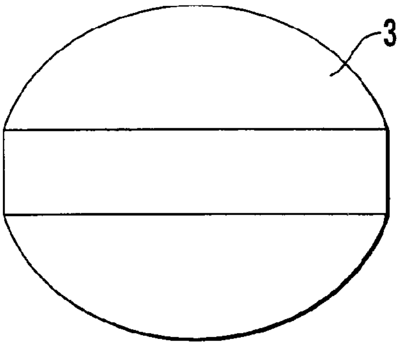

Fig. 1a

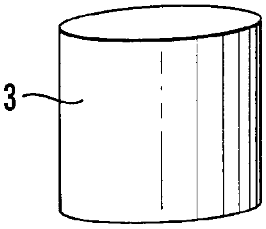

Fig. 1b

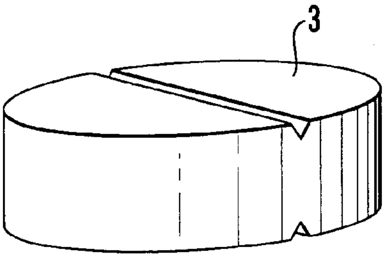

Fig. 1c

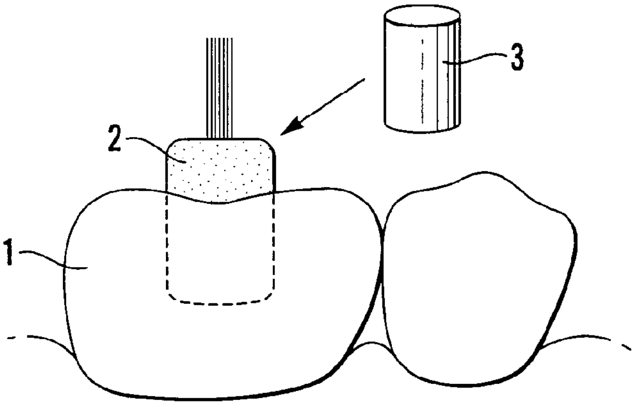

Fig.2a

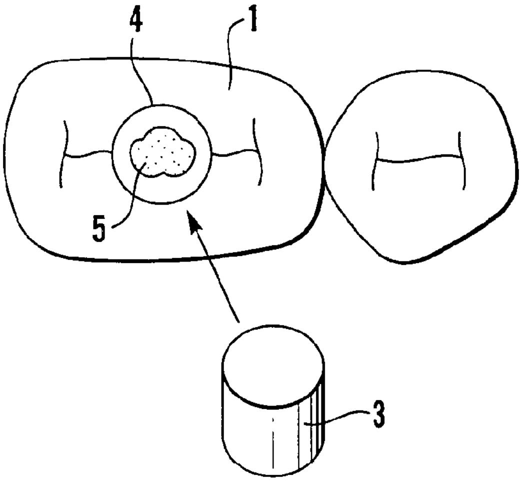

Fig.2b

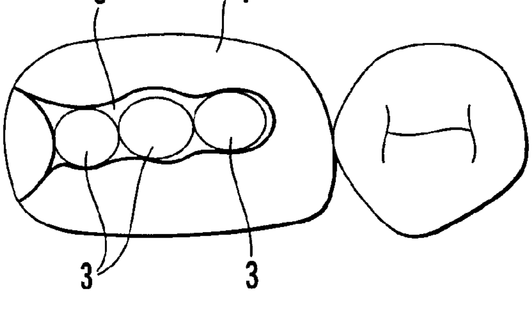

Fig.2c

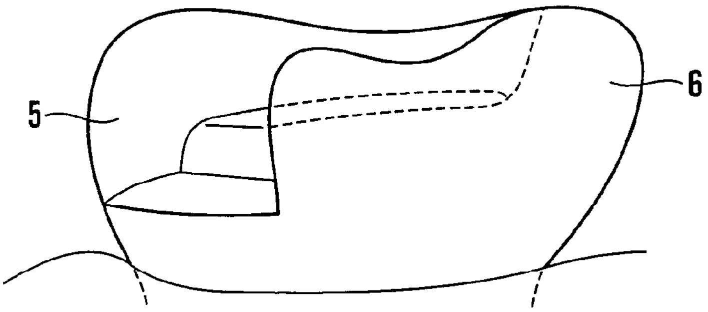

Fig.3a

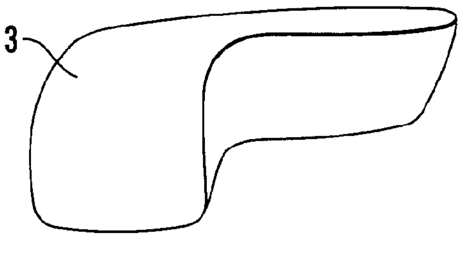

Fig.3b

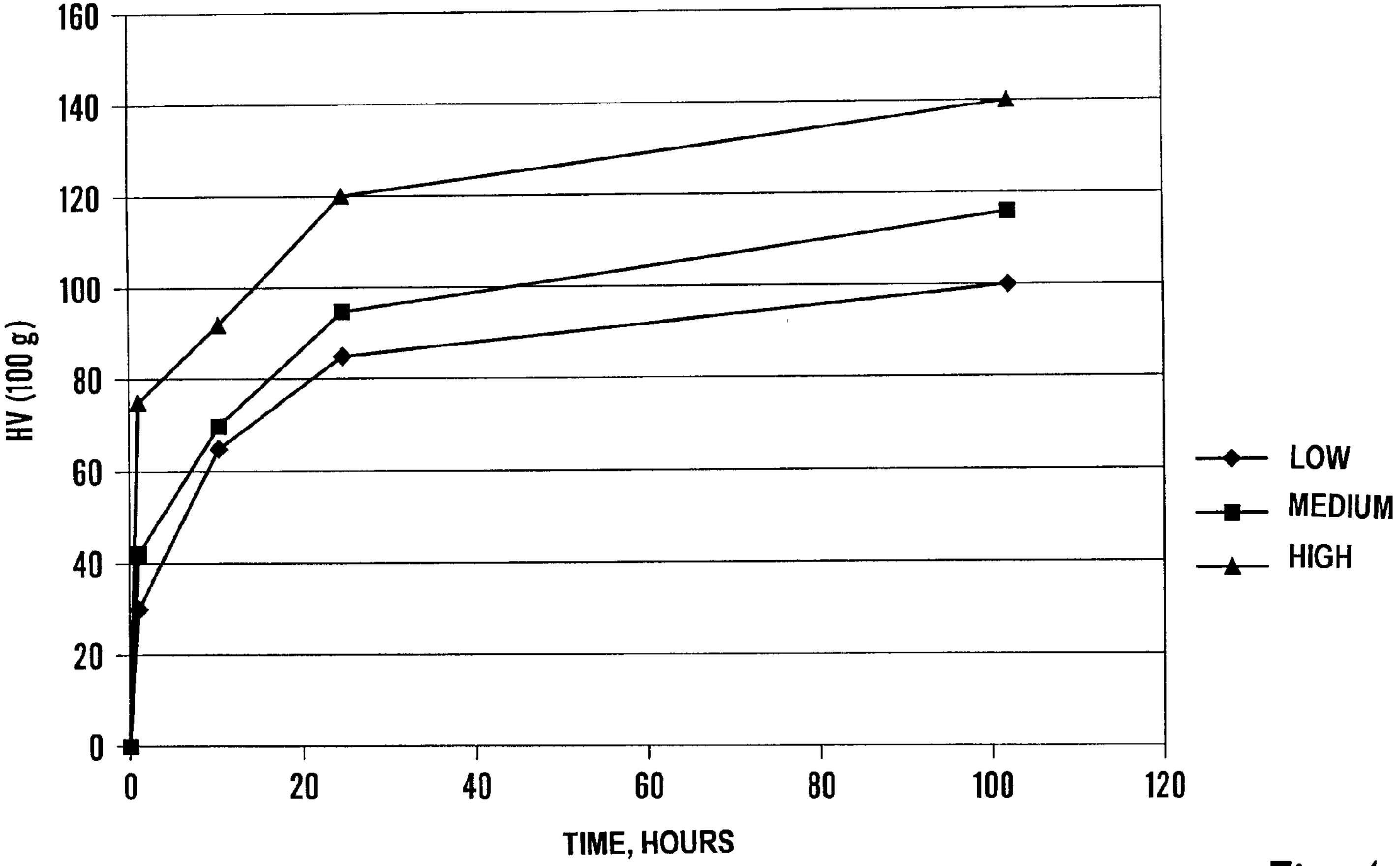
160
140
120
100
80
60
40
20
0
HV (100 g)
0
20
40
60
80
100
120
TIME, HOURS
LOW
MEDIUM
HIGH

Fig.4

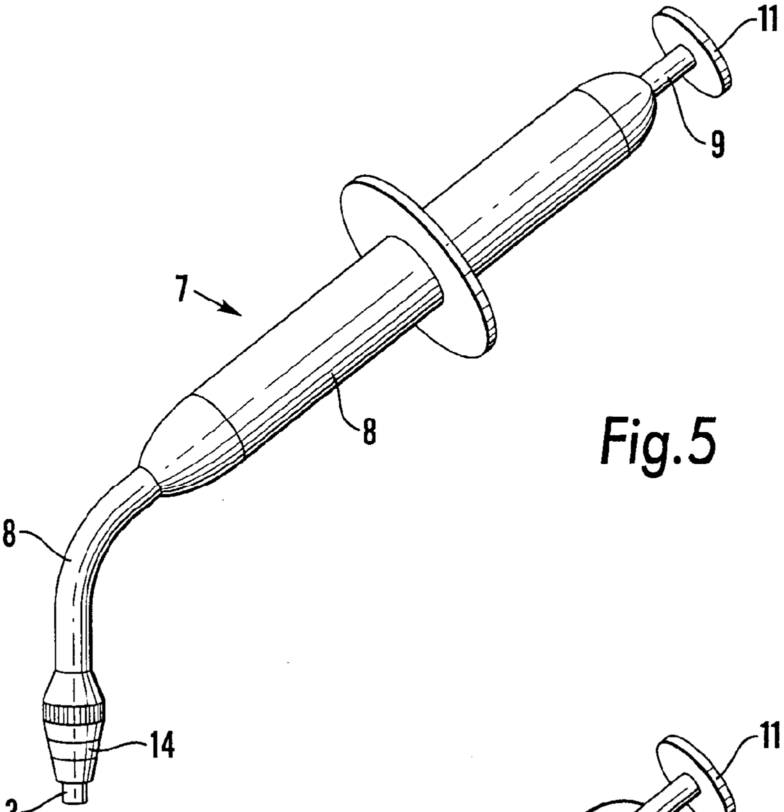

Fig.5

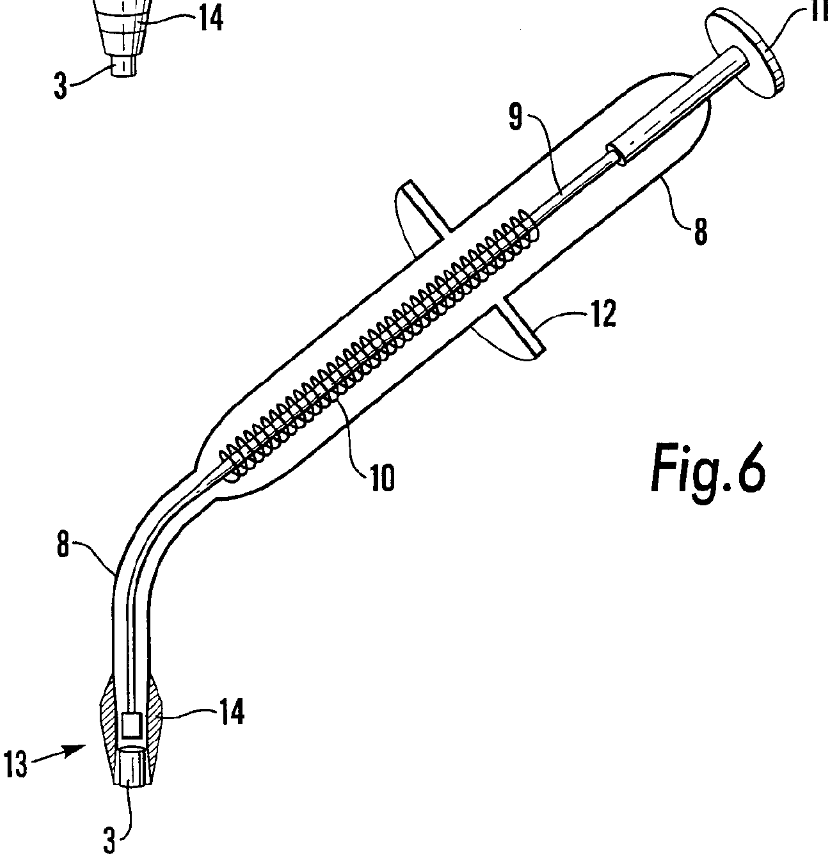

Fig.6

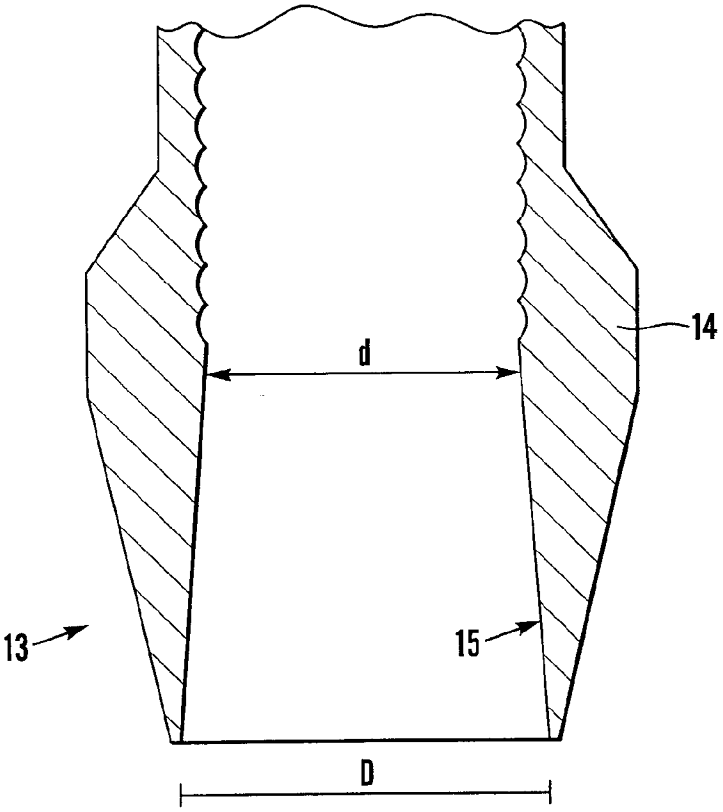

Fig.7

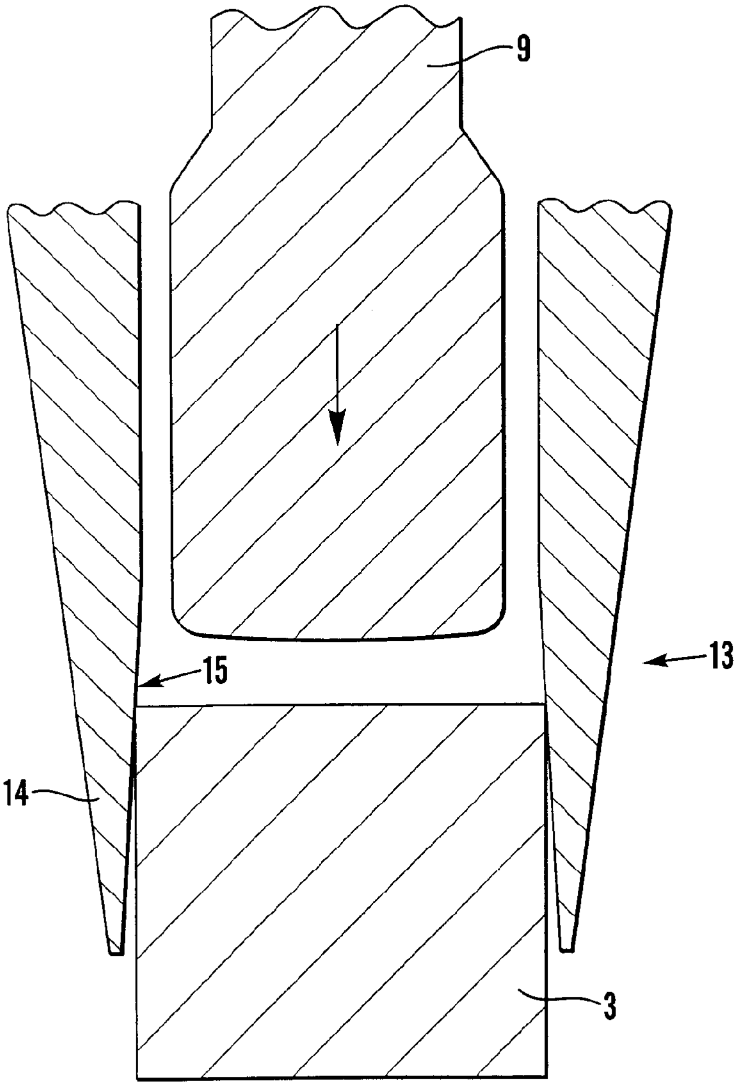

Fig.8

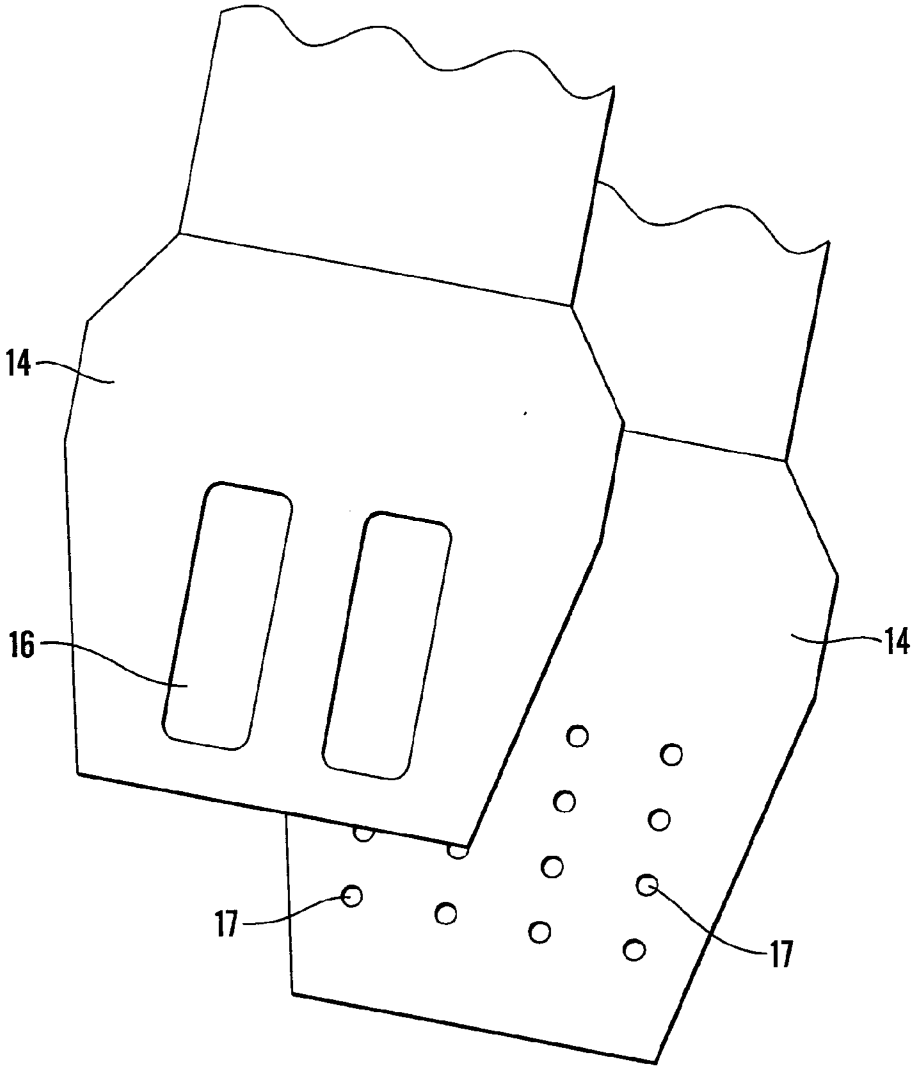

Fig.9

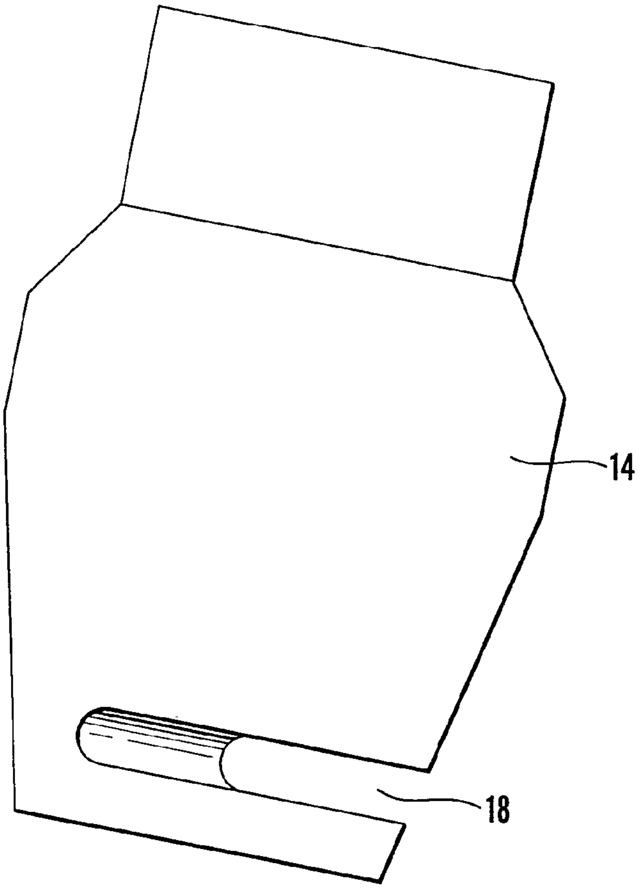

Fig. 10

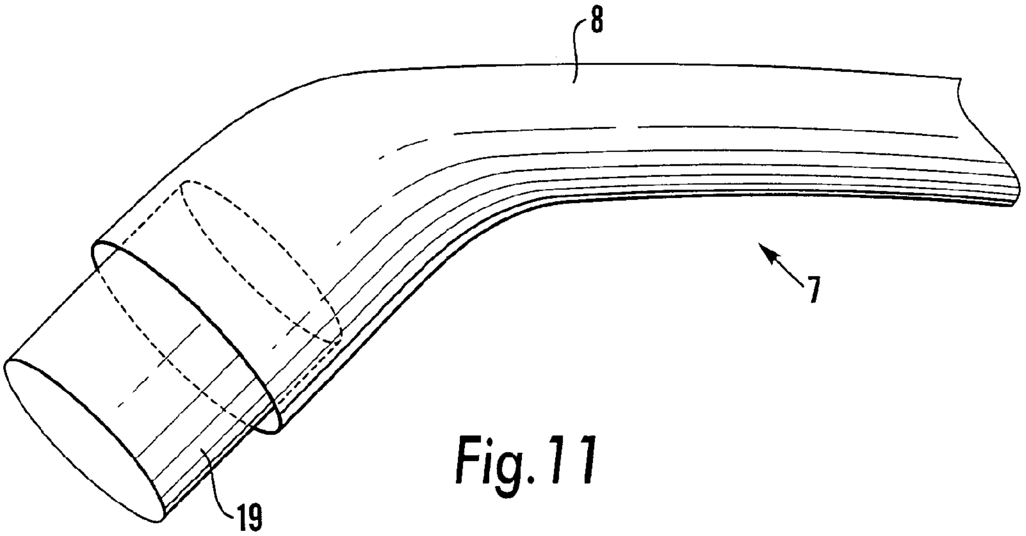

Fig. 11

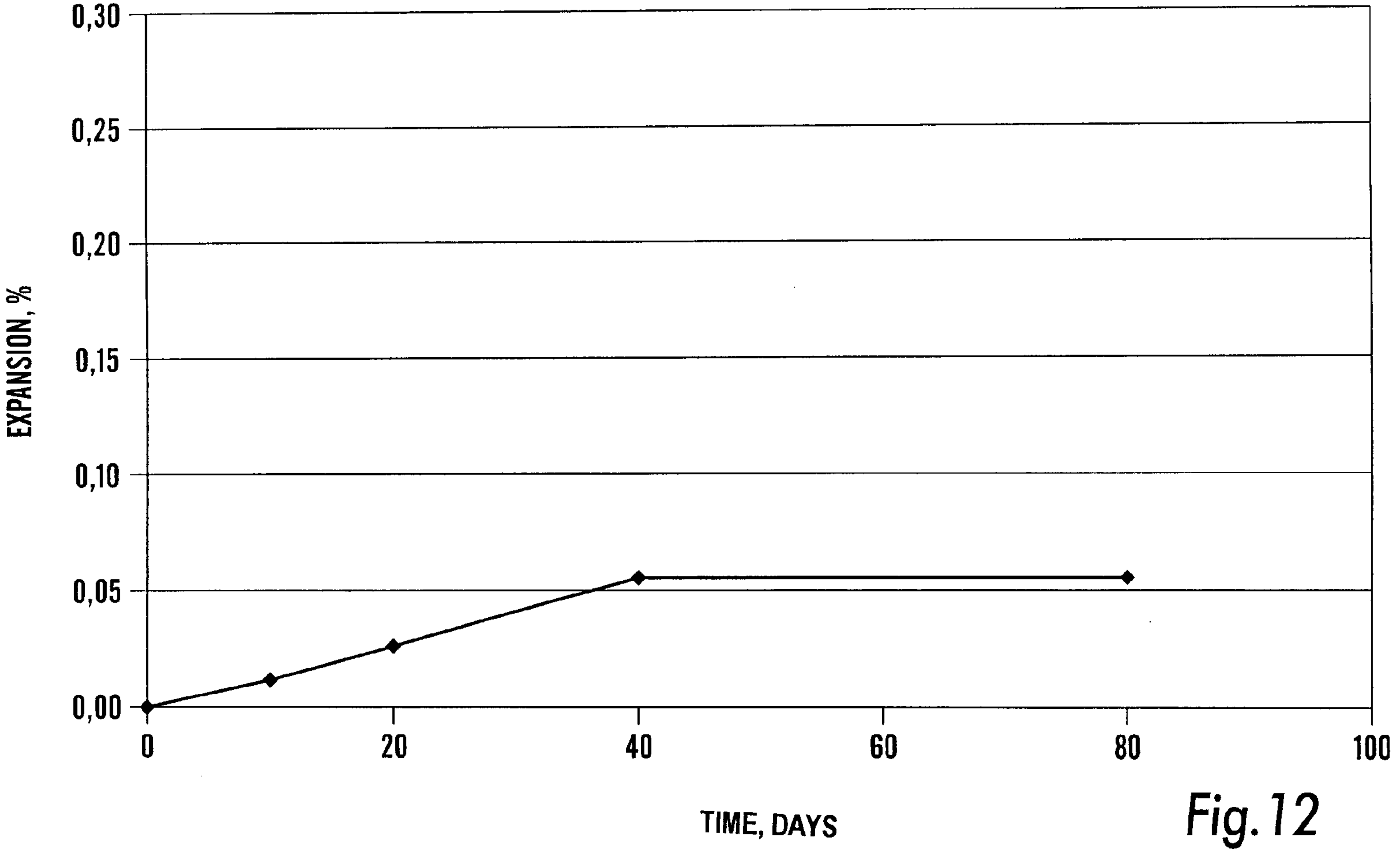
0,30
0,25
0,20
0,15
0,10
0,05
0,00
EXPANSION, %
0
20
40
60
80
100
TIME, DAYS

Fig. 12