

GrandiSO

SCIENTIFIC PRODUCT INFORMATION

VOCO
THE DENTALISTS

VOCO – THE DENTALISTS

VOCO, the family-run, independent Cuxhaven business, has set new standards in the development of innovative products with intensive research and development work for almost 30 years now. With the development of Grandio®SO, another chapter will now be added to this success story.

The BMBF project “Monomer-free-based, nano-composites as biocompatible materials for dental filling materials and prosthodontics” that ran from 2000-2003 provided the basis for the know-how in the development of filling composites. The groundbreaking findings of this research project led to the development of the first nano-hybrid composites in the world: Grandio®. Seven more years of research and development work in the VOCO laboratories as well as cooperation with over 150 universities and research facilities around the world are now represented by our new restorative: Grandio®SO.

Quality made in Germany

In 1994, VOCO was one of the first businesses able to show a certified quality assurance system (EN ISO 9001/EN ISO 13485/Standard 93/42 EEC Annex II). The approximately 20 employees in our quality control department guarantee that you always receive our products in the unvaryingly high quality that you rightfully expect from us.

Innovations for dental health

Certified quality “Made in Germany” is created at our 22,000 m² premises here in Cuxhaven. Research, production and administration under one roof ensure that communication between the individual departments is swift and collaboration intensive. Thus we are able to set standards in the development of innovative dental products. VOCO – the Dentalists.



Aerial view of company headquarters at Cuxhaven on the North Sea coast.



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GrandioSO – State Of The Art The latest technology “Made in Germany”

Nano-technology in dental materials

The word nano-technology is on the tip of everybody's tongue nowadays. It is used every day as a key term in technology and also in marketing. Nano-technology is such a multifaceted and expansive field, that a more detailed explanation of the nano-materials used in dental materials is wise.

The prefix “nano” initially just describes particles that are approximately 1-100 nano-meters in size. In other words, this is 0.000000001-0.0000001 meters. Since these numbers are also difficult to imagine, a comparison with two spheric objects that most of us are familiar with is helpful: The size of a nano-filler compared to a football is the same as a football to the earth (Fig. 1).



Fig. 1: Nano-particles compare to a football like a football compares to the earth. The size of a nano-particle thus corresponds to that of approximately 500 atoms. From a biological point of view this equals the size of the smallest bacteria or largest known enzymes.

Nano-particles are thus very small particles. Why is the use of such small particles advantageous? In order to answer this question, the function of filler particles in dental materials should be examined first. Composites consist of two substantial components: a resin and the fillers. During the polymerisation reaction the resin forms a three-dimensional network in which the filler particles are embedded. The fillers themselves primarily add strength to the composite – the inorganic filler is significantly harder than the organic network. A maximised filler content is therefore advantageous for good physical properties like strength and stability.

An additional advantage of higher filler contents is the reduced shrinkage. The organic components of the composite converge in the course of the polymerisation reaction and form

the three-dimensional network: The material experiences volumetric shrinkage. This, however, only affects the organic components. The rule of thumb is that the higher the portion of inorganic filler is, the lower the shrinkage.

In theory, only the percentage of filler must thus be increased to improve the material properties. This is not so easy in reality, however. Not only maximum stability stands in the forefront of the development of filling composites. Other parameters, such as sculptability, polishability and aesthetics, also play a pivotal role. Fillers of different sizes offer diverse advantages and disadvantages:

Macro-fillers with a diameter of 10 or more micro-meters lead to poor polishability, since whole macro-fillers can be torn out during polishing. The remaining craters cause a high surface roughness, which also impairs the aesthetics through varying reflection behaviour. The tensile strength is additionally rather low due to the relatively high inhomogeneity between organic and inorganic components. An advantage of the macro-fillers is that they do not strongly affect the viscosity of the material. Moreover, macro-fillers contribute to the material being non-tacky, so that it does not stick to the instrument.

Micro-fillers with a diameter of approx. 1-5 μm do not negatively affect the gloss. Additionally, the distribution of organic and inorganic components becomes more homogenous. The disadvantages of macro-fillers do not exist here. Micro-fillers, however, present a different problem: The so-called surface-volume ratio increases as the size of the particle decreases. Due to the extremely enlarged surface and thus contact surface to the surrounding resin, admixing micro-fillers to a composite always causes an increase in the viscosity. From a certain level onwards, the mixture becomes too stiff to permit modelling by the dentist. For this reason, the filler content is limited to approximately 80% in micro-hybrid composites.

Nano-fillers, with a diameter of 1-100 nm, possess fascinating properties, since particles of this size do not always behave as expected. In the continuation of the series macro-, micro-, nano-filler, one should expect, e.g., that the viscosity continues to increase when nano-fillers are added. The opposite, however, is the case: Nano-fillers behave like liquids to a certain degree. While micro-filled resin with a filler content of 40% has a high viscosity, a resin filled with the same concentration of nano-particles remains liquid (Fig. 2).

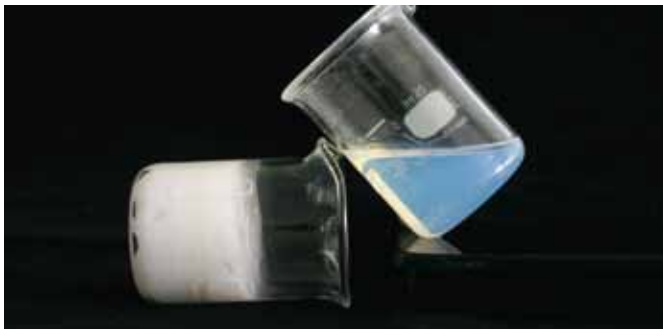


Fig. 2: A resin with 40% micro-fillers (left) behaves like a paste whereas a nano-filled resin with the same filler degree (right) still behaves like a liquid

The previously existing upper limit of approx. 80% filler content can thus be exceeded. The shrinkage is reduced and the stability further increased.

Again, the theory appears quite simple. Why then, did it take until the beginning of this millennium to develop nano-hybrid materials? The answer is based on the difficulty of manufacturing and particularly isolating particles in this very small dimension. Nano-particles are characterised by an even higher surface-volume ratio than micro-fillers. A consequence of this very high ratio is the so-called agglomeration. Nano-scaled fillers can relatively easily be manufactured by flame pyrolysis of silicium tetrachloride. The product of this process is pyrogenic silica.

Pyrogenic silica consists of small spheres with a diameter of less than 100 nm, but these spheres adhere to one another and agglomerate to form larger particles (Fig. 3). In turn, these particles have a diameter of more than 100 nm, whereby the above-mentioned positive properties of the nano-particles are lost. VOCO has succeeded in preventing this agglomeration process by providing the individual nano-fillers with a coating. A filler content of more than 85% can only be realised with the knowledge of this technology.

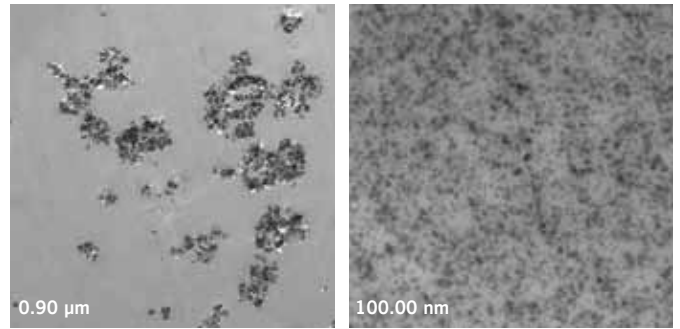


Fig. 3: Agglomerated nano-particles made by flame pyrolysis (left) and isolated nano-particles in resin (right)

The coating of the nano-fillers is responsible for additional positive properties. An organic-inorganic hybrid-compound serves as the coating material. This can take part in the polymerisation reaction of the resin, so that a firm chemical bond between nano-fillers and the surrounding matrix exists after curing. In addition to the obvious advantages relating to the stability gained from the higher cross-linking in the three-dimensional network, this is also an advantage with respect to possible risks.

The GrandioSO concept

GrandioSO is a nano-hybrid composite. Hybrid-composites are materials, in which fillers of different sizes have been added. In the case of nano-hybrids, these are micro- and nano-fillers. The larger fillers can arrange themselves to establish a more or less space-filling, sphere packing. The interstices that thereby develop are occupied by nano-fillers. It is possible to obtain a very homogeneous distribution of resin and filler in this way (Fig. 4). This is also indispensable for the excellent physical properties of GrandioSO. The composition of GrandioSO is structured as follows:

Filler:

- Glass ceramic filler with an average particle size of 1 μm
- Functionalised silicon dioxide nano-particles with a size of 20-40 nm
- Pigments (iron oxide, titanium dioxide)

Resin:

- BisGMA, BisEMA, TEGDMA

In addition, camphorquinone is used as a photocatalyst and butylated hydroxytoluene (BHT) as a stabiliser.

The combination of filler particles allows GrandioSO to have a filler content of 89% w/w.

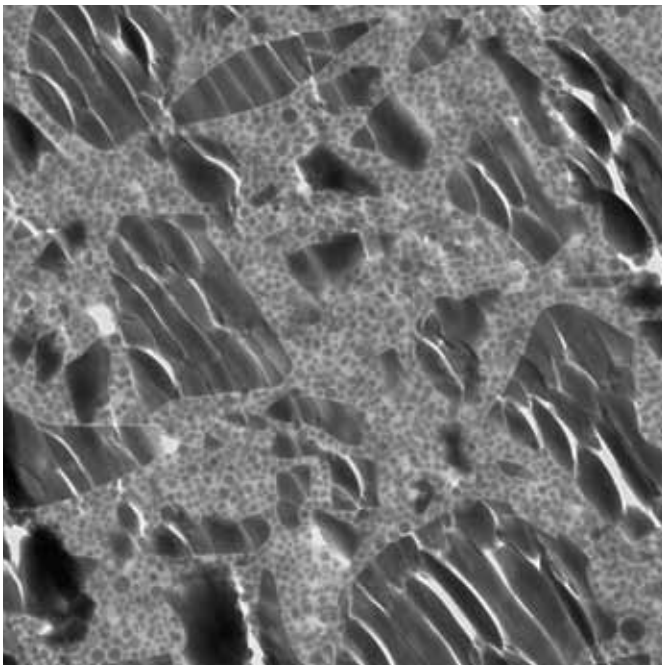


Fig. 4: Transmission-electron microscopy of GrandioSO. Homogenous distribution of nano- and micro-fillers in the resin matrix (Behrend, 2010)

Technical profile + Indications

GrandioSO		
Filler content % w/w (% v/v)	89 (73)	DIN 51081
Modulus of elasticity	16650 MPa	ISO 4049
Thermal expansion coefficient (α)	27.3	Fraunhofer Institut ISC
Shrinkage	1.61%	analogous Watts et al.
3-point flexural strength (24 h, 37 °C water storage)	187 MPa	ISO 4049
3-point flexural strength after thermocycling (3000 cycles, 5°/55°C)	158 MPa	ISO 4049
4-point flexural strength	139 MPa	University of Erlangen
Compressive strength	439 MPa	analogous ISO 9917
Edge strength	134 N	University of Manchester
Creep (7 d, 37 °C water storage)	0.36%	University of Manchester
Permanent set (7 d, 37° C water storage)	0.03%	University of Manchester
Water solubility	< 0.1 $\mu\text{g} / \text{mm}^3$	ISO 4049
Water absorption	12 $\mu\text{g} / \text{mm}^3$	ISO 4049
Surface hardness (Micro-Vickers hardness)	211 MHV	University of Rostock
Abrasion (200,000 cycles)	18 μm	ACTA 3-body
Surface gloss (polished with Dimanto, 5000 rpm)	84 GU	DIN 67530
Surface roughness R_a	0.045 μm	University of Dublin
Curing depth (800 mW/cm ²)	2.8 mm / 20 s	ISO 4049
Resistance to ambient light	4'30''	ISO 4049
Adhesion to enamel with Futurabond DC	29 MPa	University of Tanta
Radiopacity	320 %AI	ISO 4049

Indications

- Class I to V restorations
- Reconstruction of traumatically damaged anteriors
- Faceting of discoloured anteriors
- Correction of shape and shade for improved aesthetic appearance
- Locking, splinting of loosened anteriors
- Repairing of veneers
- Restoration of deciduous teeth
- Core build-up under crowns
- Composite inlays

GrandioSO – Stress Optimised

GrandioSO – Physical parameters regarding marginal integrity

In contrast to amalgam and glass ionomer cements, all composites shrink during the setting reaction. This is one of the reasons why it is mandatory to adhesively bond composites to the tooth substance. The development of marginal leakage, which could lead to the development of secondary caries if it is present for an extended period of time, can only be prevented with strong, intact bonding. All factors must be considered in the development of composite-based restoratives to counteract the development of marginal leakage in the long-term. You have to look at all factors that lead to tensile, compressive or shear forces on the adhesive bond. In the past, this was primarily attached to one value: the volumetric shrinkage. According to the theory, a composite exerts less stress on the cavity walls if the degree of shrinkage is low. This approach alone, however, has shortcomings, since other factors also affect the stress levels. This will subsequently be discussed in detail.

Shrinkage

The cause of shrinkage lies in the formation of the three-dimensional polymer network during the polymerisation. Only the resin portion of the composite contributes to the shrinkage here. Modern nano-hybrid composites, such as GrandioSO, offer a great advantage: The use of nano-fillers permits the development of composites with higher filler contents. The increasing viscosity with higher filler contents limits the maximum filler degree to approx. 80% w/w for micro-hybrid composites. With a micro-hybrid composites, the material becomes too firm to handle if the filler content is increased above this limit. This is different with the use of nano-fillers. Nano-fillers behave like a liquid to a certain degree. A content of 50-60% isolated nano-fillers does not significantly affect the consistency of the material. In GrandioSO, a total filler content of 89% w/w could be achieved by using nano-fillers. From a reversed viewpoint, this filler content means there is only 10% resin found in the composite (approx. 1% photo-catalyst, stabilisers and pigments). Only 10% of the material thus shrinks during the curing reaction, which leads to significantly reduced volumetric shrinkage, especially in direct comparison to micro-hybrids.

Shrinkage stress

The relevance of volumetric shrinkage has recently been expanded through the discussion of shrinkage stress. Shrinkage is a value that is given in volume per cent. Pure volumetric shrinkage on bonded surfaces is, however, not possible in clinical reality. A tensile force occurs on the bonding material from the shrinkage in clinical reality. This tensile force is also identified as shrinkage stress. The measuring methods for the magnitude of this tensile force vary greatly. Optical and mechanical, static and dynamic procedures have been developed. The measurement of very large increments is common in all procedures. In many measurements, test specimens are fabricated in sizes that do not correspond to the volume of composites for the application in the layer technique. Furthermore, there are always opposing cavity walls connected with an increment in these test procedures, a procedural method the layering technique is employed to especially prevent. Nevertheless, a look at these measured values is useful, since they at least permit a comparison of diverse materials, even when the amount of the measured shrinkage force is higher than it is in clinical reality.

Modulus of elasticity

The shrinkage stress represents a static load for the adhesive layer. A bond, however, is not only subject to this static load, dynamic loads also occur daily. Mastication represents the most important dynamic load in this context. Powerful forces affect a restoration every day during the chewing process. To what extent these forces are evenly discharged over the restoration is primarily determined by the modulus of elasticity, the E-modulus. This parameter describes the deformation behaviour of materials during loading. The closer the elasticity behaviour of the restorative is to the behaviour of natural tooth substance, the better the distribution of the occurring forces is in the total "tooth system". The dependence of the volume of chewing stress on the E-modulus of filling materials was analysed by Asmussen et al. (2008). The result of this study is displayed in Figure 5.

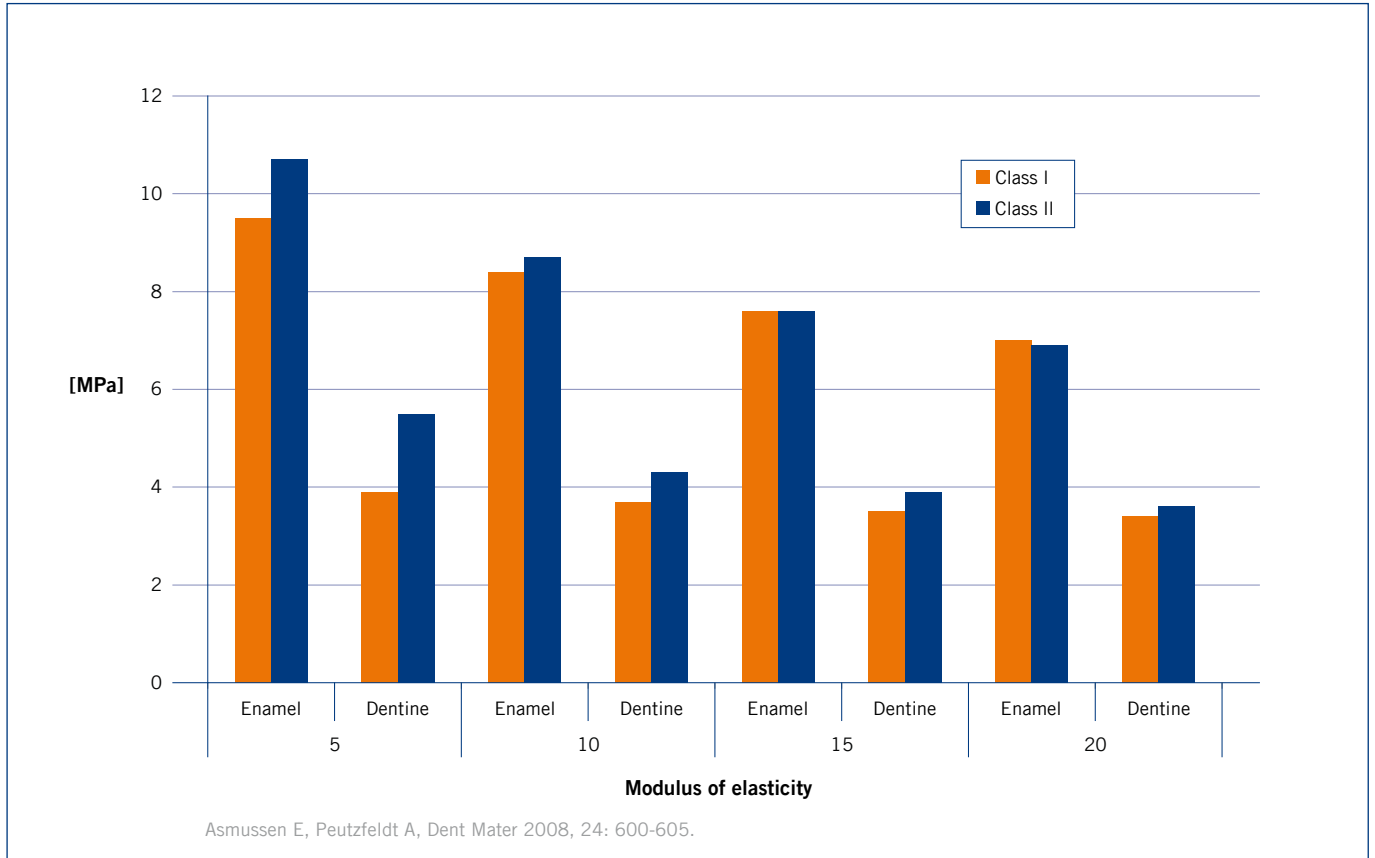


Fig. 5: Dependence of the chewing stress (y-axis) on the E modulus of restoratives (x-axis) on enamel and dentine in Class I and II fillings.

As can be gathered from the graphic, the load on the adhesive bond declines with increasing E-modulus (or better an E-modulus closer to the E-modulus of the tooth) of the material. A low E-modulus, which provides slight advantages with respect to the static stress, since it can compensate for occurring shrinkage forces through elastic deformation, thus has a negative effect under daily chewing load. Most composites have an E-modulus of 8-12 GPa (flowable composites have an even lower one). These values are significantly lower than the values for the natural tooth substance.

Evaluations

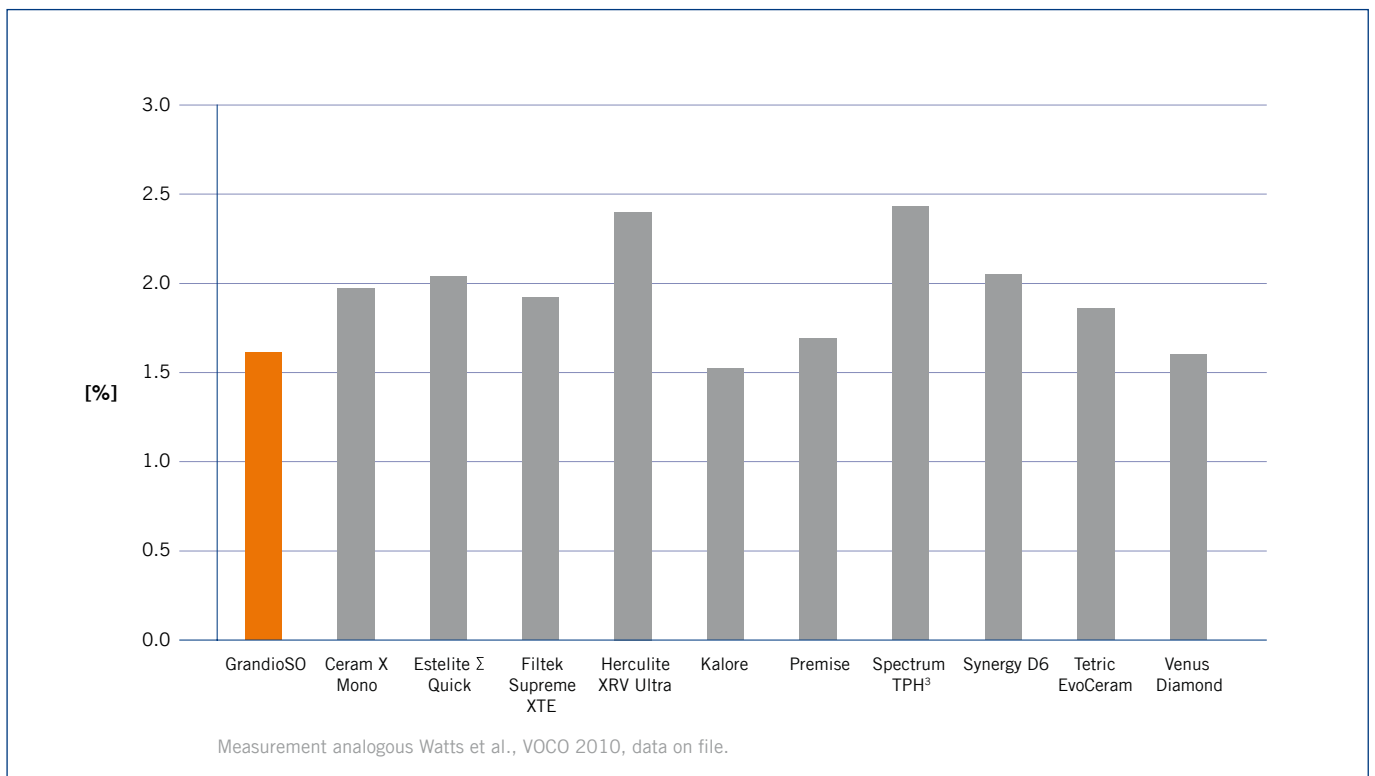
Shrinkage

Measurement procedure

The volumetric shrinkage during polymerisation was determined according to the “bonded disc” method described by Prof. Watts (University of Manchester).^[1-3] For this, a discoidal test specimen made from composite material with a diameter of approx. 8 mm and a height of approx. 1 mm was exposed to a polymerisation light (Celalux 2, Softstart, VOCO) from underneath for a total of 40 seconds. The polymerisation shrinkage was recorded with a sensor from the opposite side (top) over a period of 30 minutes.

Results

With a volumetric shrinkage of only 1.61%, GrandioSO is in the top group of the restorative composites tested.



Volumetric shrinkage [%] of the analysed composites during light-polymerisation.

Literature

- [1] Kim und Watts, 2004.
- [2] Watts und Cash, 1991.
- [3] Watts und Marouf, 2000.

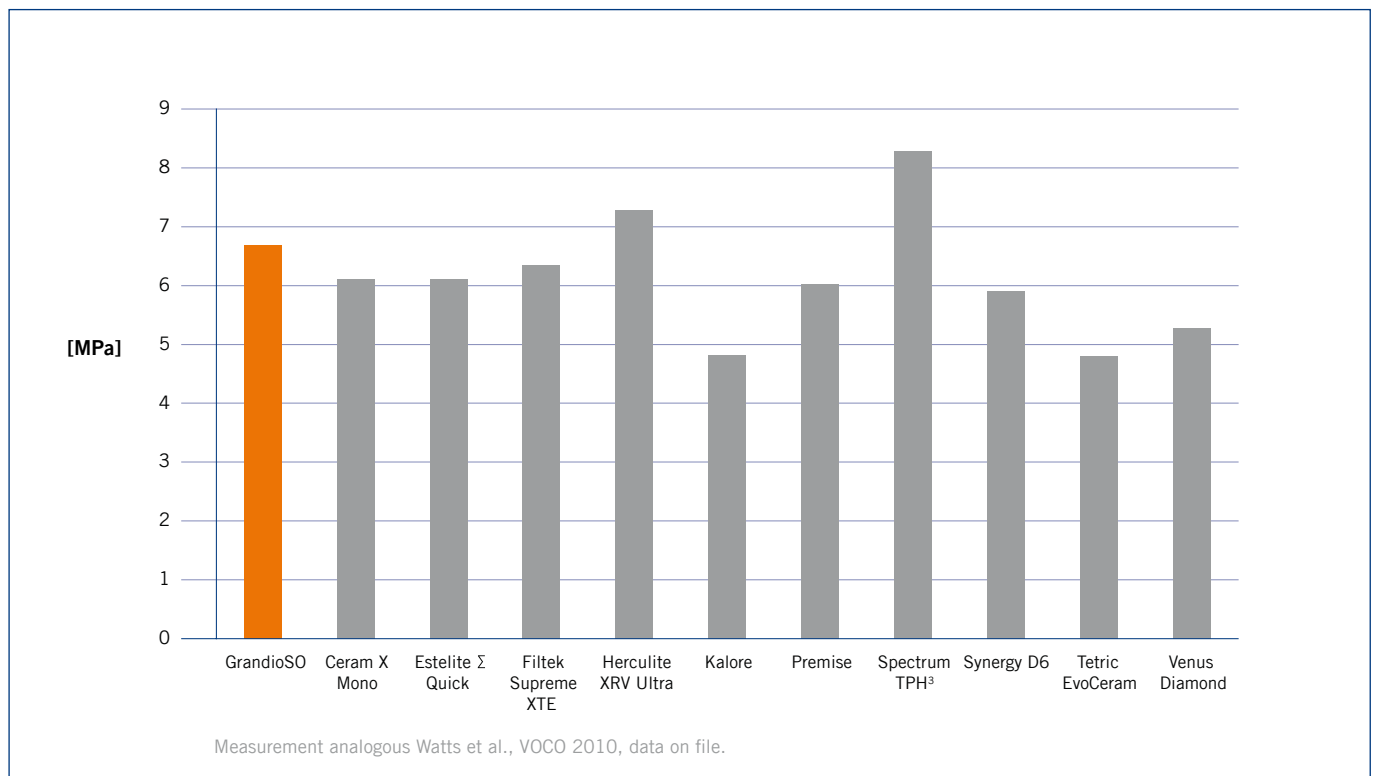
Shrinkage stress

Measurement procedure

The shrinkage stress after polymerisation was determined according to the so-called “bioman” method developed by Prof. Watts (University of Manchester).^[1-2] A cylindrical specimen of the material with a height of 0.75 mm and a diameter of 8 mm was polymerised from underneath through a fixed glass plate for 40 seconds. A steel cylinder was connected to the measuring apparatus on the surface of the composite, which was first roughened with a sandblaster. The force exerted on this cylinder was then recorded over a period of 30 minutes and the resulting polymerisation tension of the composite subsequently calculated.

Results

The shrinkage range is about 6 MPa for most of the tested materials. GrandioSO has a shrinkage stress of 6.68 MPa. This is slightly higher than the values of a few of the materials compared, but the differences are minimal.



Polymerisation shrinkage stress [MPa] of tested composites.

Literature

- [1] Watts und Satterthwaite, 2008.
- [2] Watts et al., 2003.

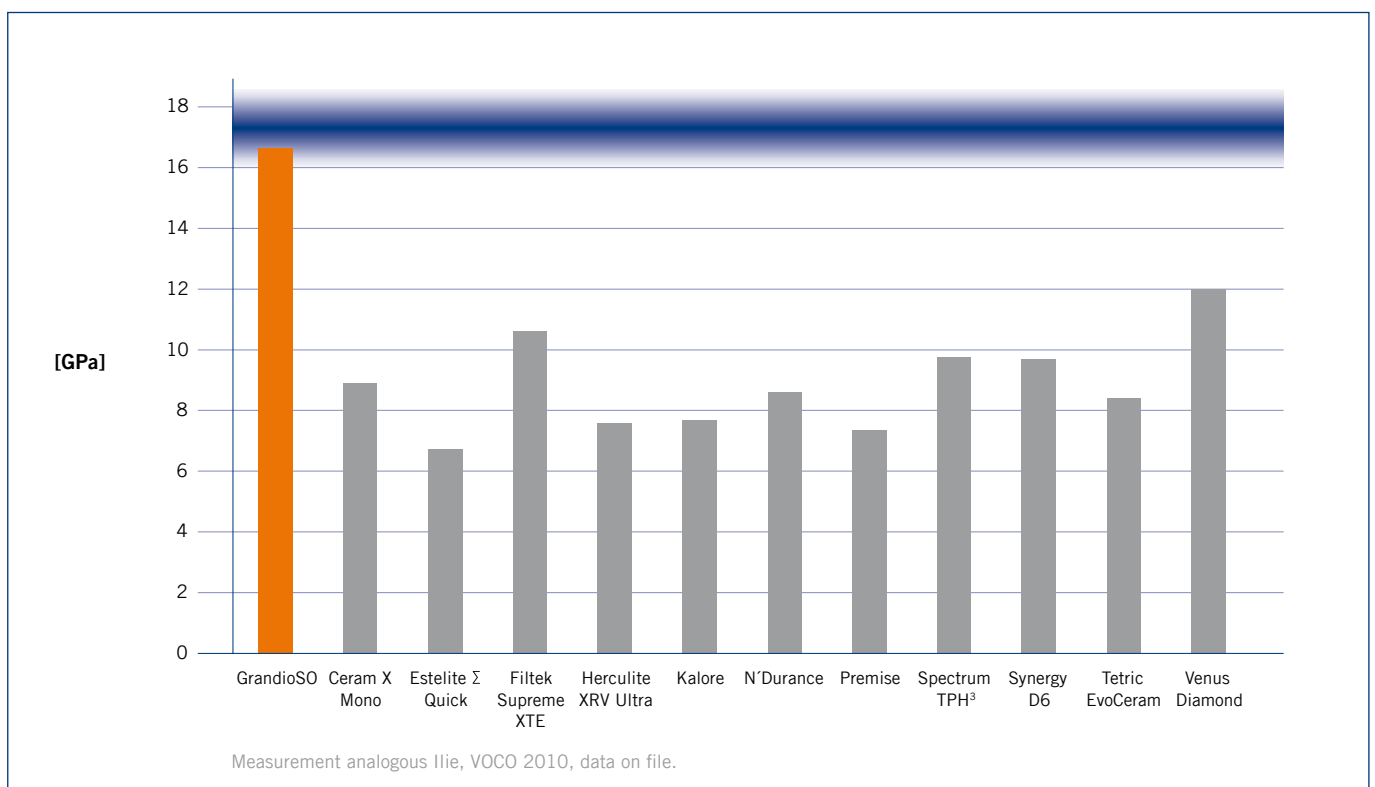
Modulus of elasticity

Measurement procedure

The E-modulus was determined from the measurements of the 3-point flexural strengths by calculating the increase in the linear range of the corresponding transverse strength gradient.^[1]

Results

With an E-modulus of 16.65 GPa, only GrandioSO exhibits an elasticity behaviour close to that of dentine. In literature, the E-modulus of dentine varies between 16.55 and 18.62 GPa (blue line).^[2] With regard to the elasticity behaviour, GrandioSO behaves exactly like the natural tooth substance.



Modulus of elasticity [GPa] of different composites.

Literature

[1] Ilie, 2004.

[2] Craig und Peyton, 1958.

Thermal behaviour

In addition to the E-modulus, there is yet another factor that is often ignored in the examination of the long-term integrity of the margins: The thermal behaviour of restoratives. Like most materials, composites expand when heated and contract when cooled. This behaviour also applies to the tooth.

With the consumption of ice cream, for example, the temperature drops on the tooth surface, which leads to contraction of the tooth and the restorative. If the contraction behaviour of the restorative is more pronounced than that of the tooth, tensile force develops on the adhesive. The degree of thermal volume change is described with the thermal expansion coefficient α . As in the examination of the E modulus, the mea-

surement of the value alone is not significant. Decisive again is the comparison to the behaviour of the natural tooth substance (enamel: $\alpha = 17$, dentine: $\alpha = 11$; Xu et al. 1989).

The extent of the temperature changes was examined in an *in vivo* study by Ernst et al. (Ernst et al. 2004). The study came to the conclusion that the consumption of hot beverages led to an average increase in temperature of the tooth to 43.8°C, while the consumption of iced water led to an average cooling to 24.2°C. The averaged initial temperature was 35.2°C. Based on the study above, the consumption of ice cream leads to a cooling down by 11°C.

Evaluations

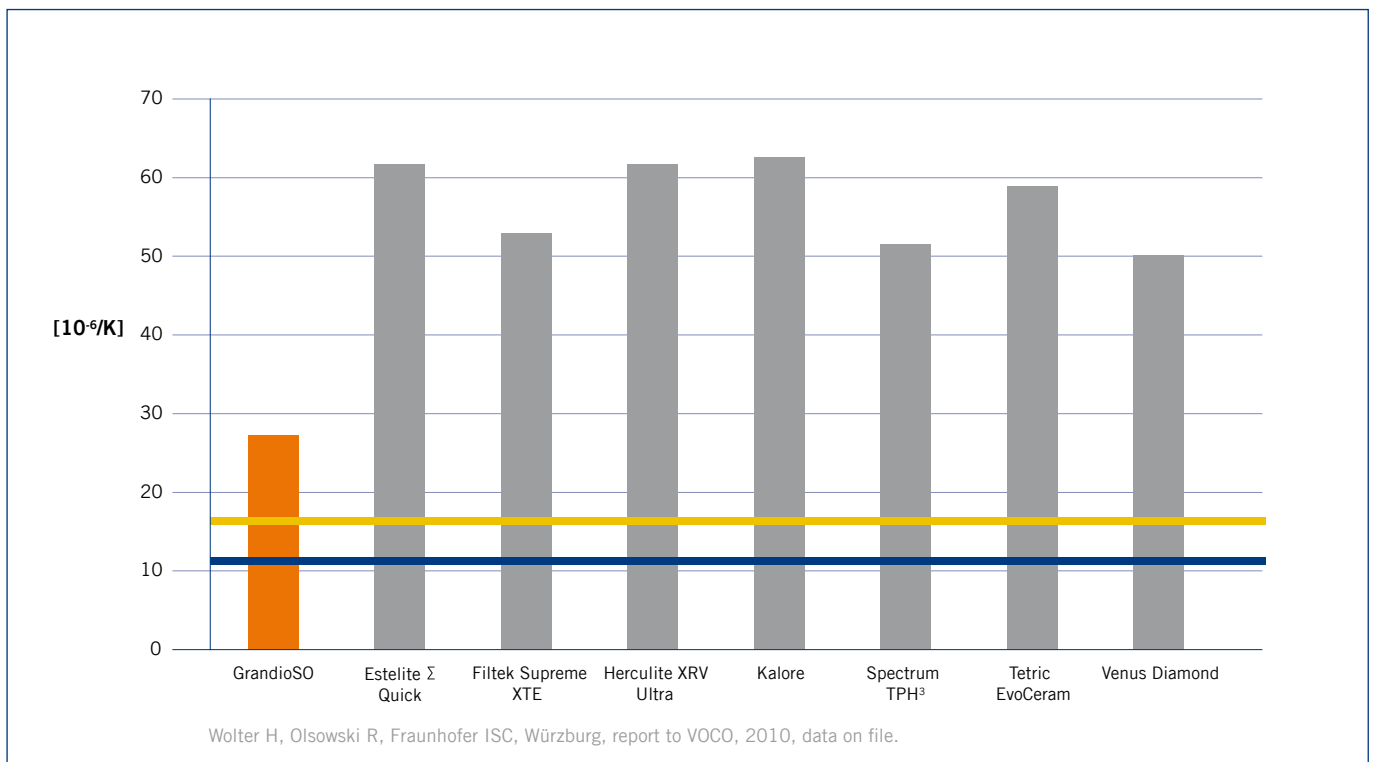
Thermal expansion coefficient

Measurement procedure

2x2x30 mm test specimens were prepared for the measurement of the thermal expansion coefficient α . The linear expansion of these rods was determined in a range of 25-50°C at a heating rate of 1 Kelvin/Minute with a connecting rod dilatometer.^[1]

Results

In literature, the expansion coefficients α of dentine and enamel are given with $10.59 \cdot 10^{-6}/K$ (blue line) and $16.96 \cdot 10^{-6}/K$ (yellow line) respectively.^[2] GrandioSO may not reach exactly these values, but its values are much closer to the expansion and contraction behaviour of the natural tooth substance than the other materials tested. The stress on the filling margins from the thermal expansion is thus reduced to a minimum.



Thermal expansion coefficient [$10^{-6}/K$] of the tested composites.

Literature

[1] Wolter, 2010.

[2] Xu et al., 1989.

Example: Thermal behaviour in Class I restoration

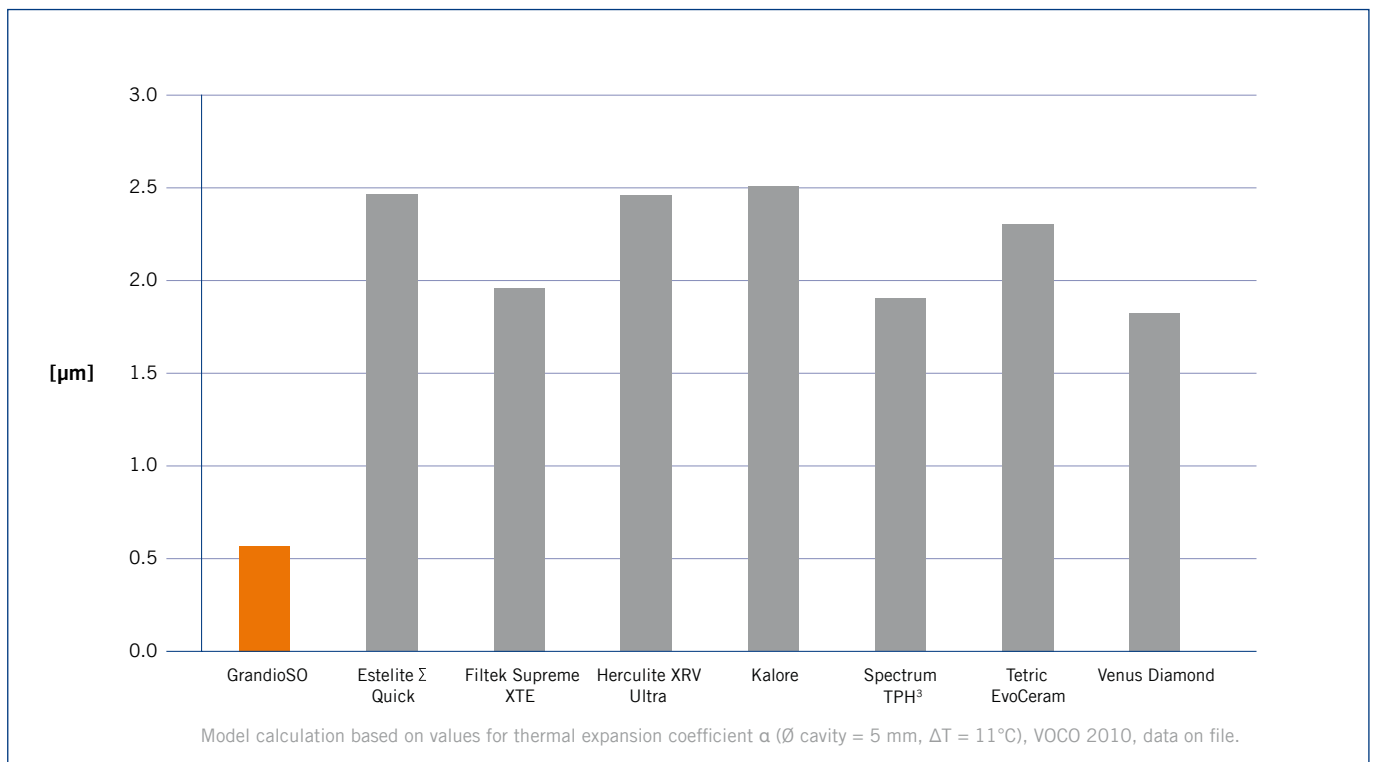
Calculation method^[1]

A Class I filling with a diameter of 5 mm was used as a basis here for the calculation of the effect of different contraction behaviour. The temperature difference was specified at 11 °C, as it was measured for the consumption of ice cream.^[2] The formula for the calculation is:

$$\Delta L = (\alpha_{\text{Enamel}} \times L_0 \times \Delta T) - (\alpha_{\text{Composite}} \times L_0 \times \Delta T)$$

Results

The figure shows by how many more micro-meters the respective restorative contracts than the surrounding enamel. The material, of course, cannot contract unimpeded due to the bond. The smaller change in the volume of GrandioSO (3-5 times lesser) leads to significantly less withdrawal forces than other materials tested. These values support long-term, intact filling margins, despite the alternating thermal loading that occurs daily.



Volume change [µm] (here: contraction) under thermal loading.

Literature

- [1] Ohring, 1995.
- [2] Ernst et al., 2004.

Summary

The long-term quality of the adhesive bond is affected by many factors. These include volumetric shrinkage, shrinkage stress, elasticity behaviour and thermal behaviour of the composite material. To achieve optimum quality, it is imperative to reduce the static loads, which occur with polymerisation. The shrinkage during polymerisation was reduced to a minimum with GrandioSO. With the development of GrandioSO, appropriate attention was also given to the shrinkage stress that arises during light-curing. An adequate layering technique when placing the filling can also positively influence these factors. The tooth-like behaviour of the restorative, which contributes to minimisation of the dynamic loads from masticatory forces and thermal influences, is also equally crucial. With an modulus of elasticity of 16.65 GPa, GrandioSO exhibits a value that is comparable to dentine (E-modulus dentine: 18.5 GPa; Willems et al. 1993). GrandioSO performed best in comparison to the other composites in the test with respect to thermal volume change. GrandioSO exhibited a huge difference to other restoratives particularly in the elasticity behaviour and thermal-related volume change.

When all factors are considered, GrandioSO offers tooth-like behaviour and thus the best prospects for long-term, intact margins.

GrandioSO – Strength Optimised

GrandioSO – Physical parameters regarding stability

Restoratives are subjected to powerful loads every day. Chewing pressure represents the most frequent and important load. This force is on average 30.6 ± 5.6 MPa (Miyaura et al., 1999), whereby the pressure on smaller contact surfaces (e.g. nut splitter) is much higher. A restorative must endure these forces without suffering any damage. To describe the stability of materials, diverse physical parameters are determined: flexural strength(s), compressive strength, edge strength, tensile strength and many others.

Flexural strength and fatigue resistance

Composites are elastic materials that deform under application of force. Flexural strength measurements examine at what point the load leads to fractures of the material. Different procedures are used for this. While only the type of load varies with 3- and 4-point transverse strength measurements, cumulative damage from the deformations is also examined in the measurement of fatigue resistance. The restorative should not only withstand one chewing cycle, but also many years of daily mastication.

Compressive and tensile strength

Compressive and tensile strength are parameters that are closely linked to transverse strength. One can say that these two values itemise the flexural strength. If a body is bent, then high compressive forces affect the material on the surface of the concave side, while tensile forces prevail on the convex side. To what extent these individual loads lead to a failure of the material is determined in these examinations.

Edge strength

The edge strength is a value that is especially important for the restoring of load-bearing cusps in the posterior range. The value describes the fracture resistance of side margins of composite test specimens and thus describes the tendency to resist chipping.

Creep and permanent set

The creep and permanent set values describe the deformation behaviour of materials under pressure. When a force affects a body, then it is initially compressed. The measure of this

compression is partially described by the E-modulus. After the initial quick compression, another, slower compression takes place in the course of the next minutes to hours. This compression goes along with creep processes inside the composite. The physical structure is reorganised in some areas to compensate for local peaks of load. A counter-process takes place as soon as the applied force is removed. An expansion takes place within a short time that leads to a recovery of 80-90%. Another, slower, relaxation subsequently takes place. But even after this relaxation time, however, the original volume value is not reached again. This difference is called permanent set. One example for this phenomenon from daily life are lane grooves.

The value describes the inelastic deformation and is therefore very important for posterior restorations. If a material cannot withstand the daily chewing loads, deformations on the occlusal surface result over the years. This, of course, affects the occlusion, so that deformations can lead to serious problems. Low creep supports long-term shape stability.

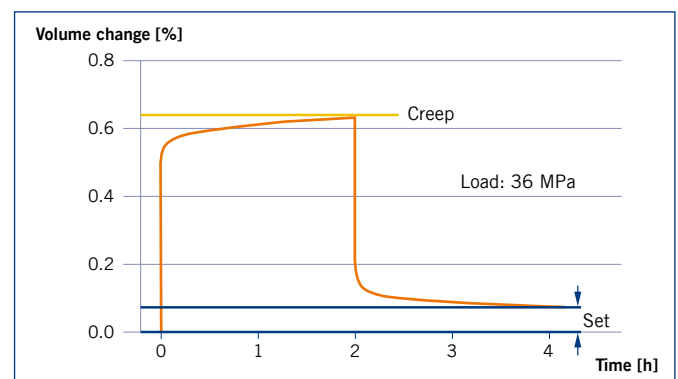


Fig. 6: Measurement of Creep and permanent set

Adhesion

The adhesion of composites naturally depends more on the adhesive system used than on the composite itself. Nevertheless, a composite must establish a good adhesive bond to the bonding material. This value alone, however, is not measurable, so that the tooth/bond/composite system is always measured in the examination.

Evaluations

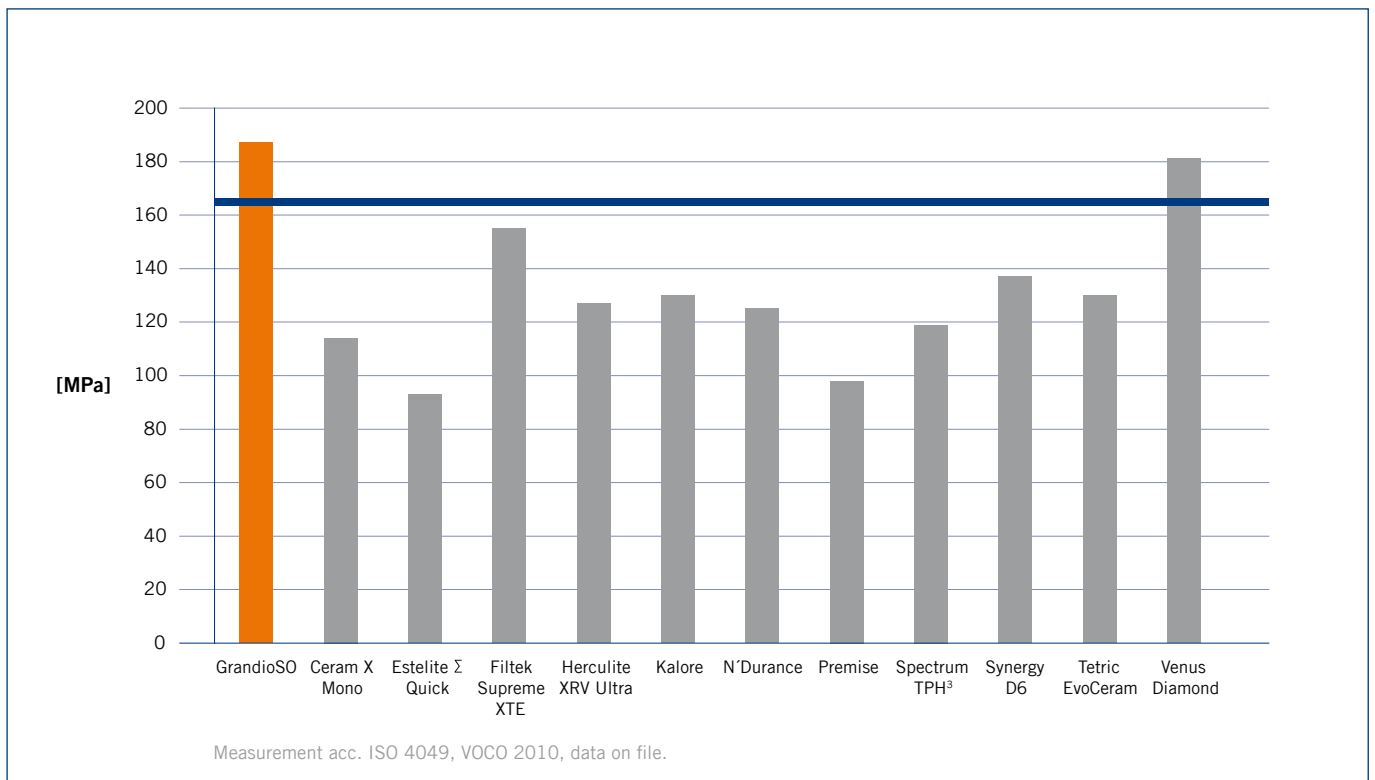
3-Point flexural strength

Measurement procedure

The procedure for the measurement of the 3-point flexural strength is described in ISO 4049.^[1] In accordance with this standard, five test specimens with the dimensions 2x2x25 mm were prepared and loaded in a force-way measuring device for a total of 0.75±0.25 mm/min. The test specimens were placed on two bars in the process, while pressure was applied to the centre from above with a third rod. The stated flexural strength is the average value at which the test specimen breaks. In the ISO Standard, a minimum value of 80 MPa is mandatory for light-curing, composite-based restoratives.

Results

GrandioSO delivered the highest value for flexural strength in this test (187 MPa). A comparison with dentine is interesting, because a flexural strength of 165.6 MPa is stated in literature (blue line).^[2]



3-Point flexural strength [MPa] of the tested composites.

Literature

[1] ISO 4049, International Organization for Standardization.

[2] Jameson et al., 1993.

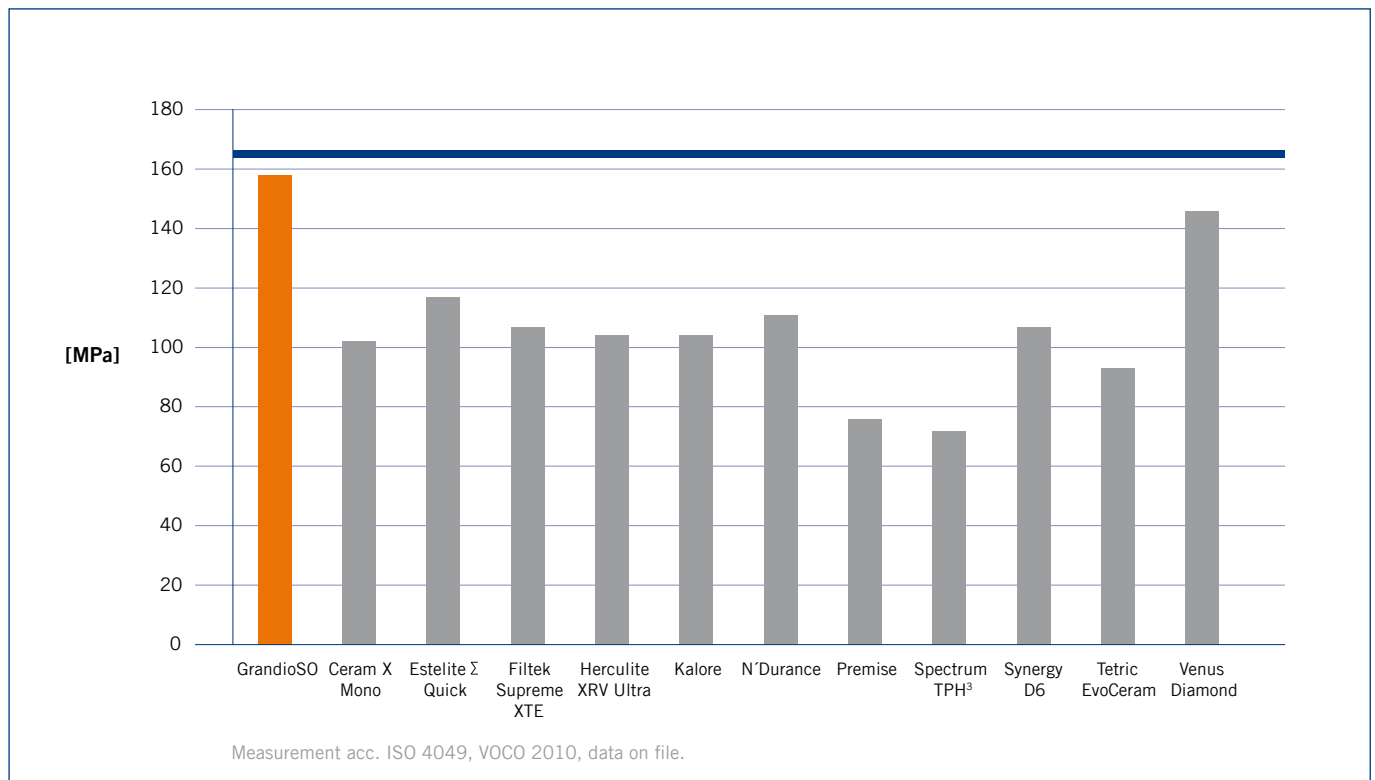
3-Point flexural strength after thermocycling

Measurement procedure

To simulate the ageing of materials, they are subjected to the so-called thermocycling. In this procedure, the test specimens are alternatively being heated to 55 °C and cooled to 5°C in a aqueous medium. This cycle was run 3,000 times in total. The 3-point flexural strength was subsequently determined as described above.^[1]

Results

As expected, the values obtained for the flexural strength were somewhat lower after the thermocycling than prior to the artificial ageing. GrandioSO also exhibited, however, the highest value here from the examined composites with a value of 158 MPa. Even after the artificial ageing, GrandioSO's flexural strength was the closest to that of dentine (flexural strength dentine: 165.6 MPa, blue line).^[2]



3-Point flexural strength [MPa] of the tested composites after thermocycling.

Literature

- [1] ISO 4049, International Organization for Standardization.
- [2] Jameson et al., 1993.

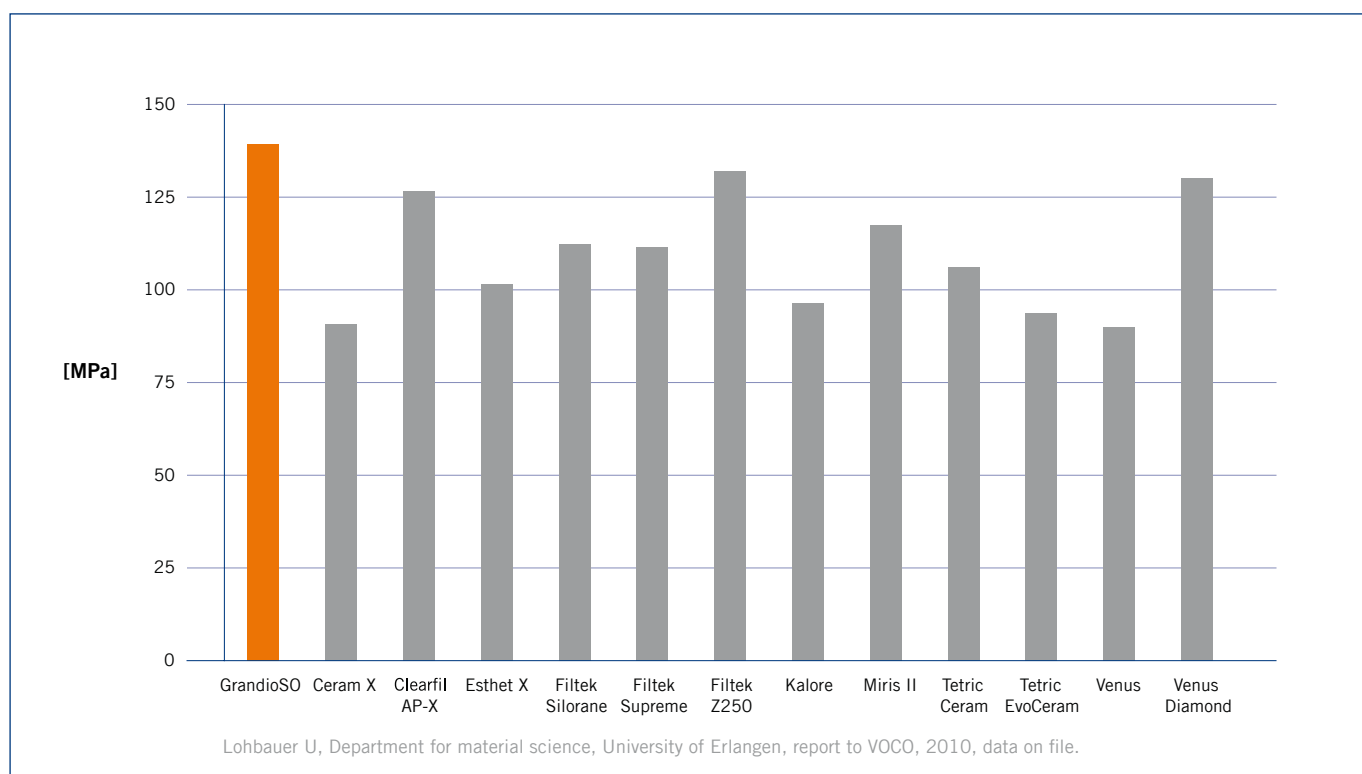
4-Point flexural strength

Measurement procedure

The 4-point transverse strength was measured in a study at the University of Erlangen.^[1] In contrast to the 3-point flexural strength measurement, the test specimen is placed on 2 supporting rollers (d = 2 mm, distance: 20 mm) and loaded in the centre on 2 loading points with cylindrical pressure fins (d = 2 mm, distance: 10 mm). The loading speed was 0.75 mm/min. As a deviation from the measurement in the 3-point procedure described in ISO 4049, here the specimens were also stored in distilled water at 37°C for two weeks prior the testing.

Results

Following this protocol, GrandioSO exhibited the highest value for the 4-point flexural strength at 139.23 MPa.



4-Point flexural strength [MPa] of different composites.

Literature

[1] Lohbauer, 2010.

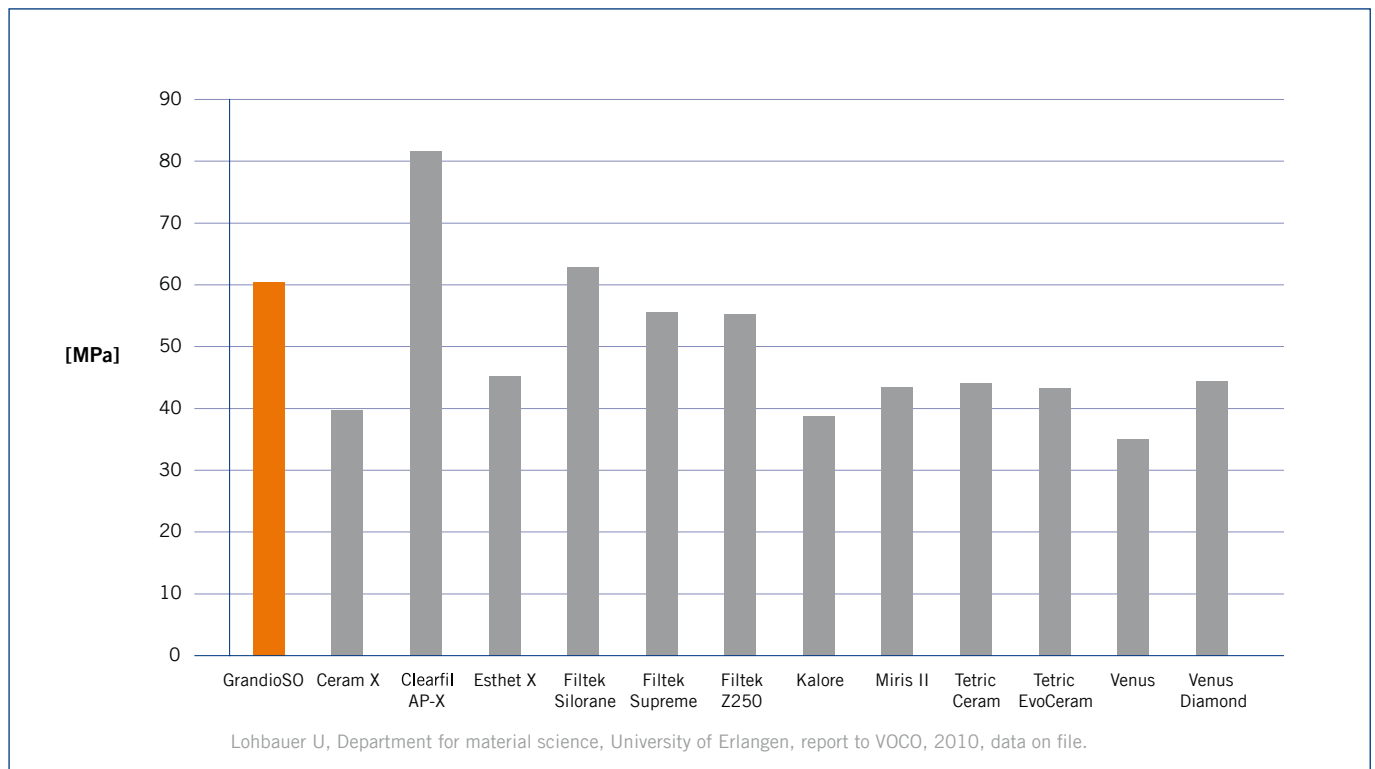
Fatigue resistance

Measurement procedure

To determine the flexural fatigue limit, test specimens were fabricated analogous to the 4-point transverse strength measurement procedure. These were then subjected to a sinusoidal load with a frequency of approx. $f = 0.5-1$ Hz in max. 10,000 cycles. The maximum stress σ_{max} was defined as 50% of the initial flexural strength. All test specimens that endured the first cycle were exposed to ever-increasing loads in the so-called staircase method.^[1]

Results

GrandioSO exhibited an excellent value of 60.5 MPa for fatigue resistance, which supports the expectation of a long-lasting retention period for the placed filling.



Resistance to fatigue [MPa] of the tested composites.

Literature

[1] Lohbauer, 2010.

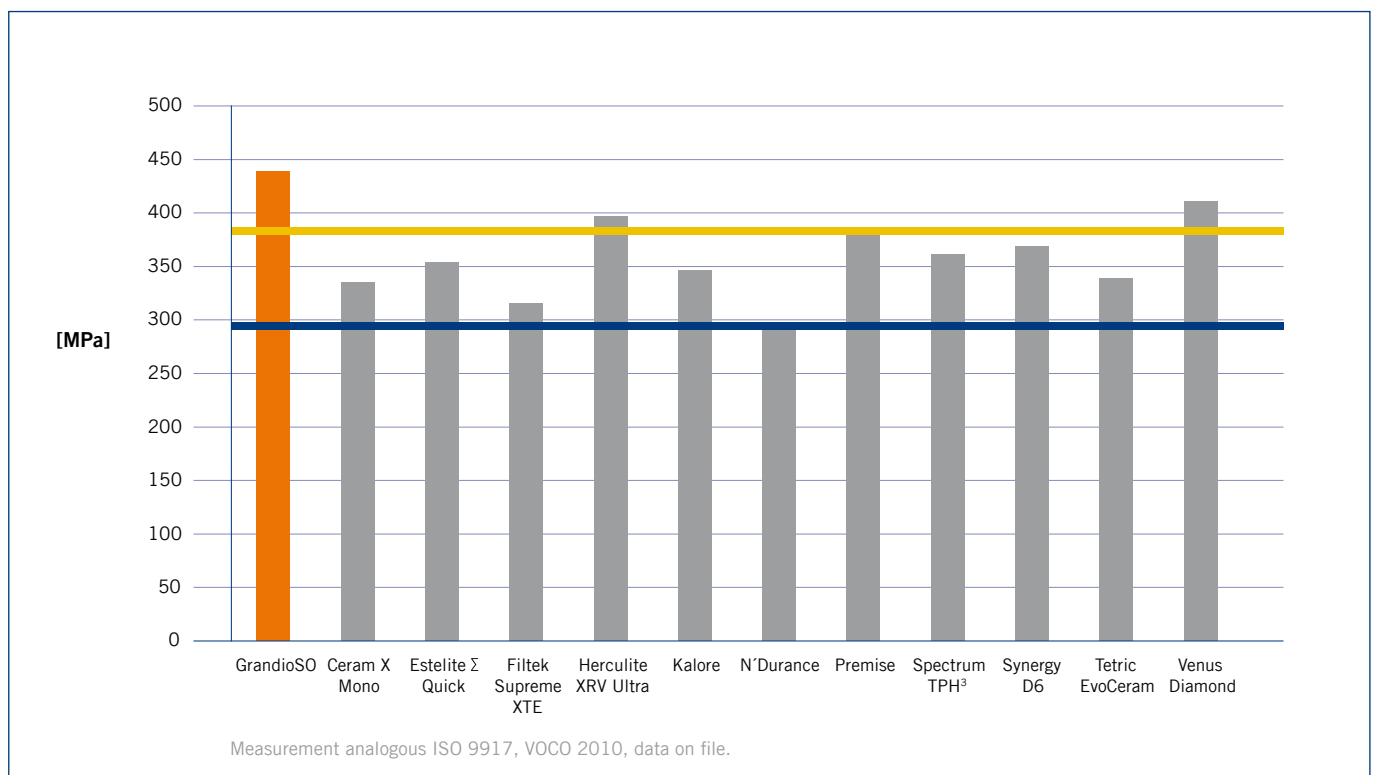
Compressive strength

Measurement procedure

The measurement of the compressive strength was conducted analogous to the procedure for cements described in ISO 9917.^[1] For this, a 6 mm high cylinder with a diameter of 3 mm was fabricated. The test specimen was subsequently loaded with a force of 50 ± 16 N/min until it failed under the applied load. The load under which the test specimen breaks is described as its compressive strength.

Results

GrandioSO achieved the highest value in this measurement with a compressive strength of 439 MPa. This means that GrandioSO has a higher compressive strength than the natural tooth substance (dentine 297 MPa^[2] – blue line; enamel 384 MPa^[3] – yellow line). GrandioSO is thus able to endure high peak loads.



Compressive strengths [MPa] of the tested composites.

Literature

- [1] ISO 9917, International Organisation for Standardization.
- [2] Craig und Peyton, 1958.
- [3] Craig et al., 1961.

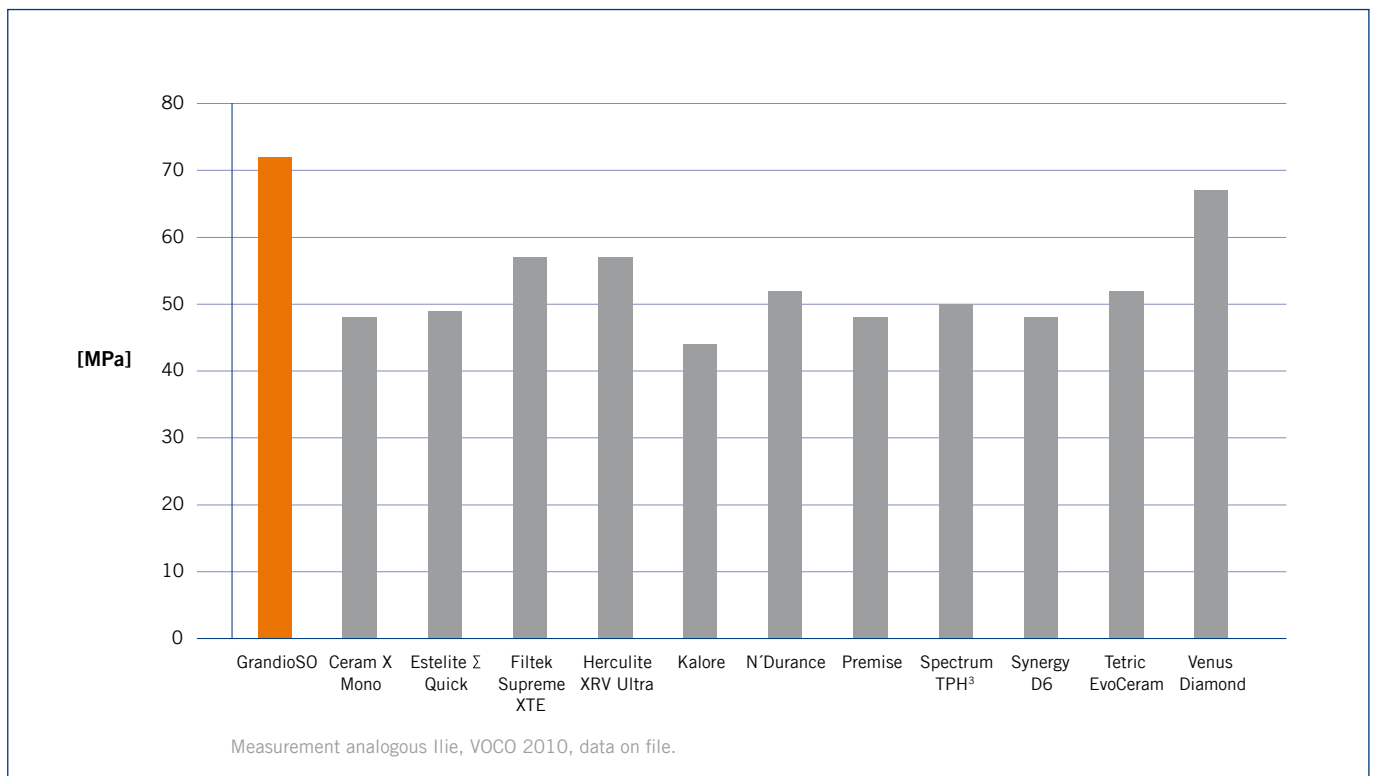
Diametral tensile strength

Measurement procedure

For the determination of the diametral tensile strength, cylindrical test specimens with a diameter of 3 mm and a height of 6 mm were fabricated.^[1] These were subsequently placed on a force-way measuring device with the longitudinal side on the metal block. The test specimens were loaded with a second metal block at a speed of 1 mm/min until they broke. The tensile strength results from the maximum force and the exact test specimen dimensions.

Results

With a value of 72 MPa, GrandioSO has the highest diametral tensile strength of all the materials tested in this measurement. GrandioSO has thus almost twice as much tensile strength as natural dentine, which has a tensile strength of 37.3 MPa.^[2]



Diametral tensile strength [MPa] of different composites.

Literature

[1] Ilie 2004.

[2] Jameson et al., 1993.

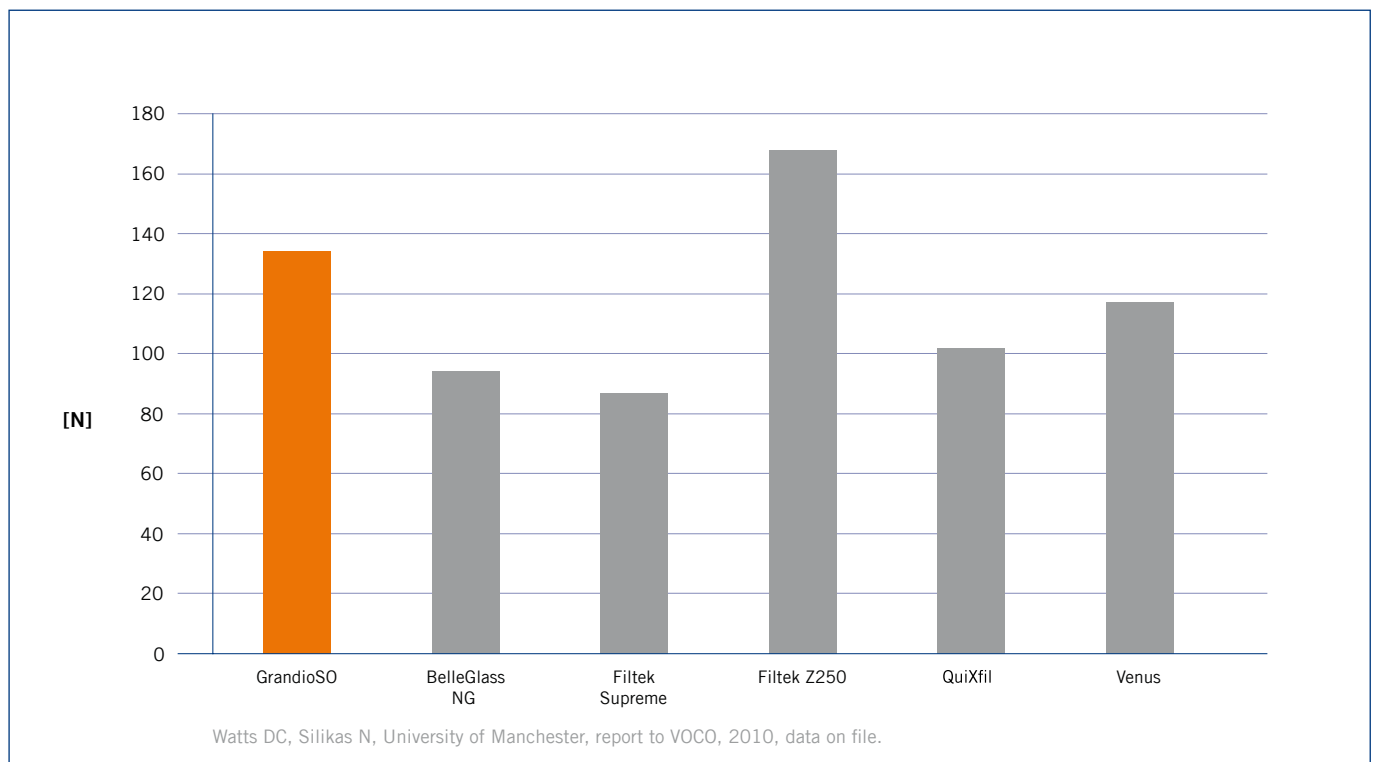
Edge strength

Measurement procedure^[1]

The edge strength was determined with a special measuring device (CK10, Engineering Systems) at the University of Manchester. Test specimens with a diameter of 12 mm and a height of 2.5 mm were fabricated and stored in water at 37 °C for 7 days. The pressure was applied with a diamond tip at a distance of 5 mm from the edge, and speed was 1 mm/min. Both chipping and a complete break were rated as an error. Detection was performed with an acoustic sensor.

Results

GrandioSO has an edge stability of 134.4 N, which represents the second best value in this study.



Edge stability [N] of diverse composites.

Literature

[1] Watts und Silikas, 2010.

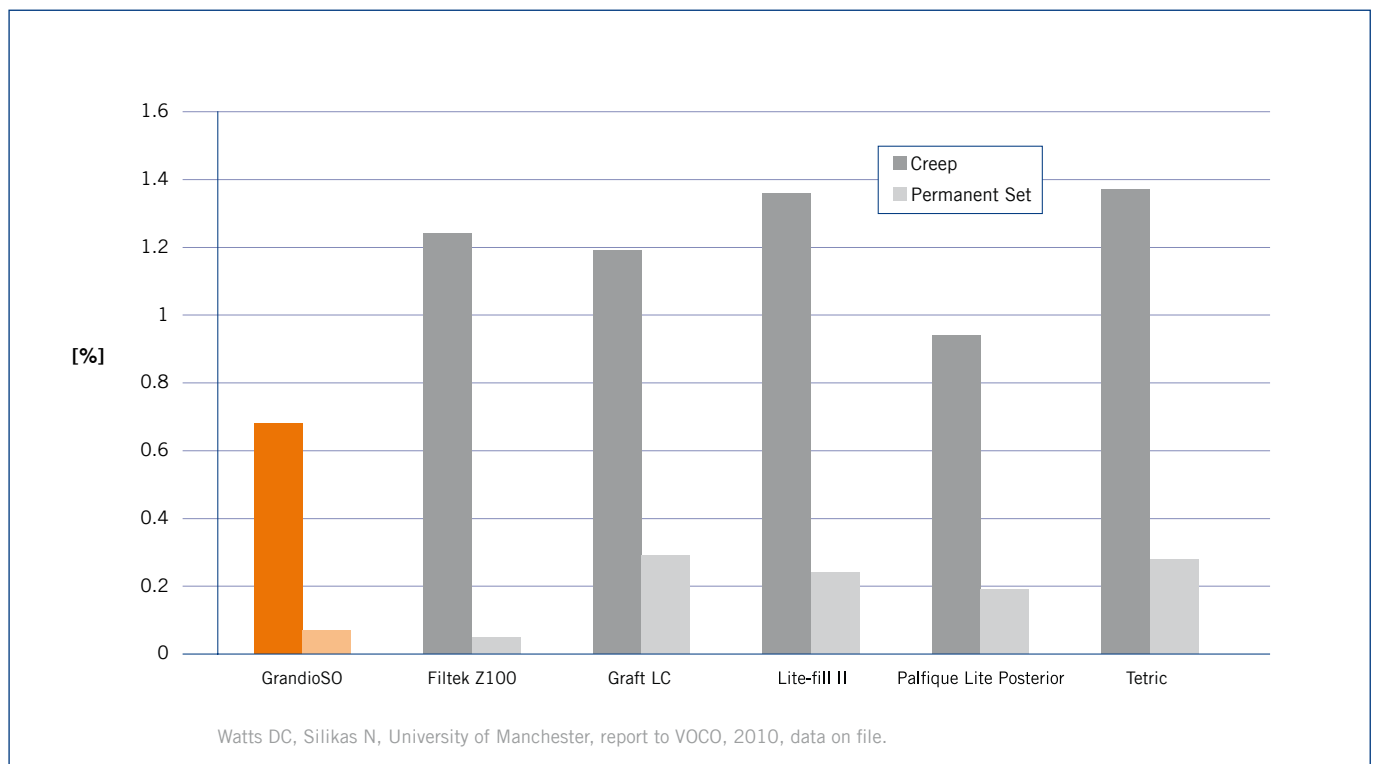
Creep

Measurement procedure

Cylindrical test specimens (6 mm long, 4 mm diameter) were fabricated and stored in dry conditions at 37 °C for 7 days before the measurement. The cylinders were thereupon loaded in a special creep measuring device for 6 hours with a force of 36 MPa, after which the test specimens were not loaded for 6 hours afterwards. The elastic deformation before the rest period is indicated by the creep, the permanent deformation after the rest period is indicated by the permanent set.^[1] The creep and permanent set values for GrandioSO were determined according to this method at the University of Manchester.^[2]

Results

GrandioSO exhibited an extremely low inelastic deformation. This is an important prerequisite for long-term shape stability of Class I and Class II restorations, which are exposed to chewing forces over years.



Creep and permanent set [%] of the tested composites.

Literature

- [1] El Hejazi und Watts, 1999.
- [2] Watts und Silikas, 2010.

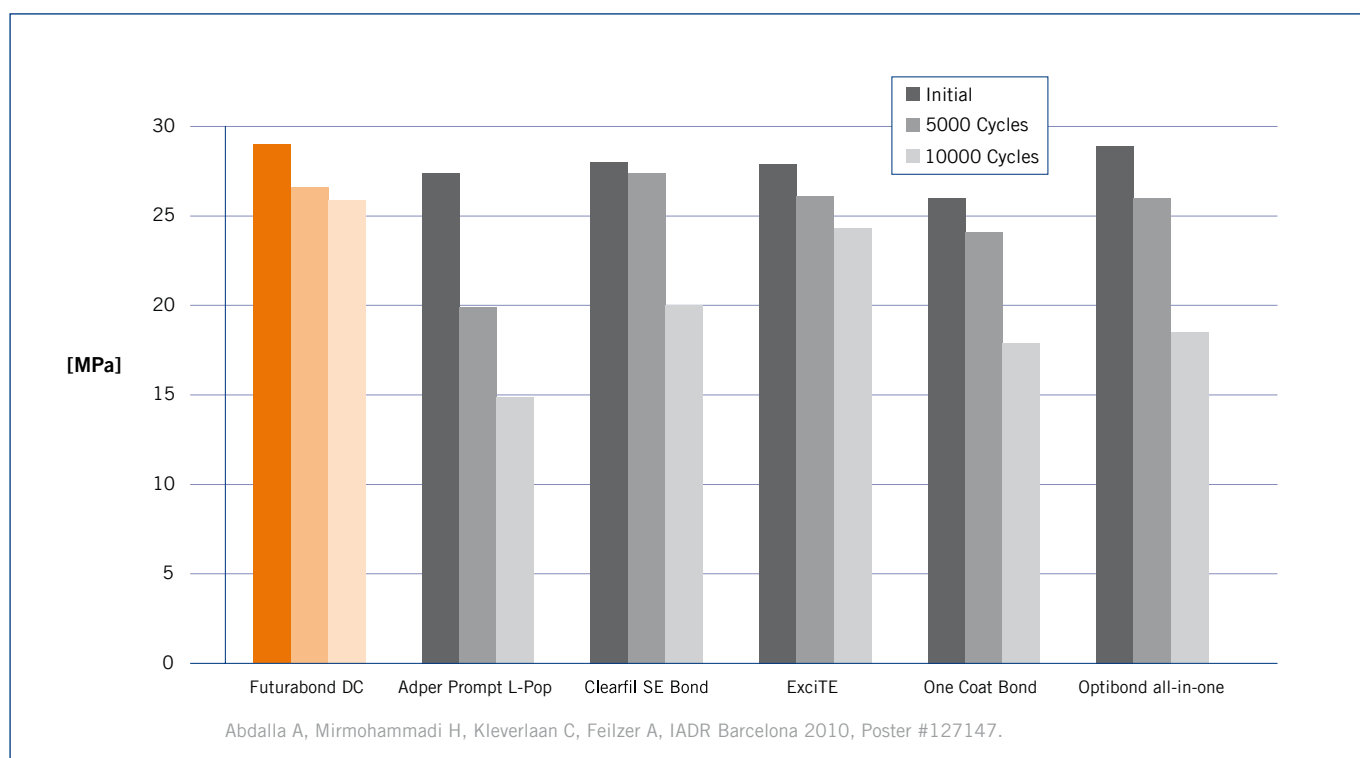
Adhesion values on enamel

Measurement procedure

Buccal and lingual surfaces of human posterior teeth were ground flat using silicon carbide abrasive paper (600-grit) and treated with different adhesive systems for this examination of the adhesion values of GrandioSO. A 2x2 mm sized increment of GrandioSO was subsequently applied. After 24 hours of storage in water, the adhesion on enamel was determined with a shear test. Additional test specimens were subjected to a shear bond test after 5000 and 10000 cycles of thermocycling respectively.^[1]

Results

GrandioSO initially offers excellent adhesion with all of the tested bonding systems. After ageing, excellent bonding strength values of 25 MPa and above were achieved with Futurabond DC (VOCO) and Excite (Ivoclar-Vivadent).



Adhesion values [MPa] of GrandioSO with different bonding materials, initial and after thermocycling.

Literature

[1] Abdalla et al., 2010.

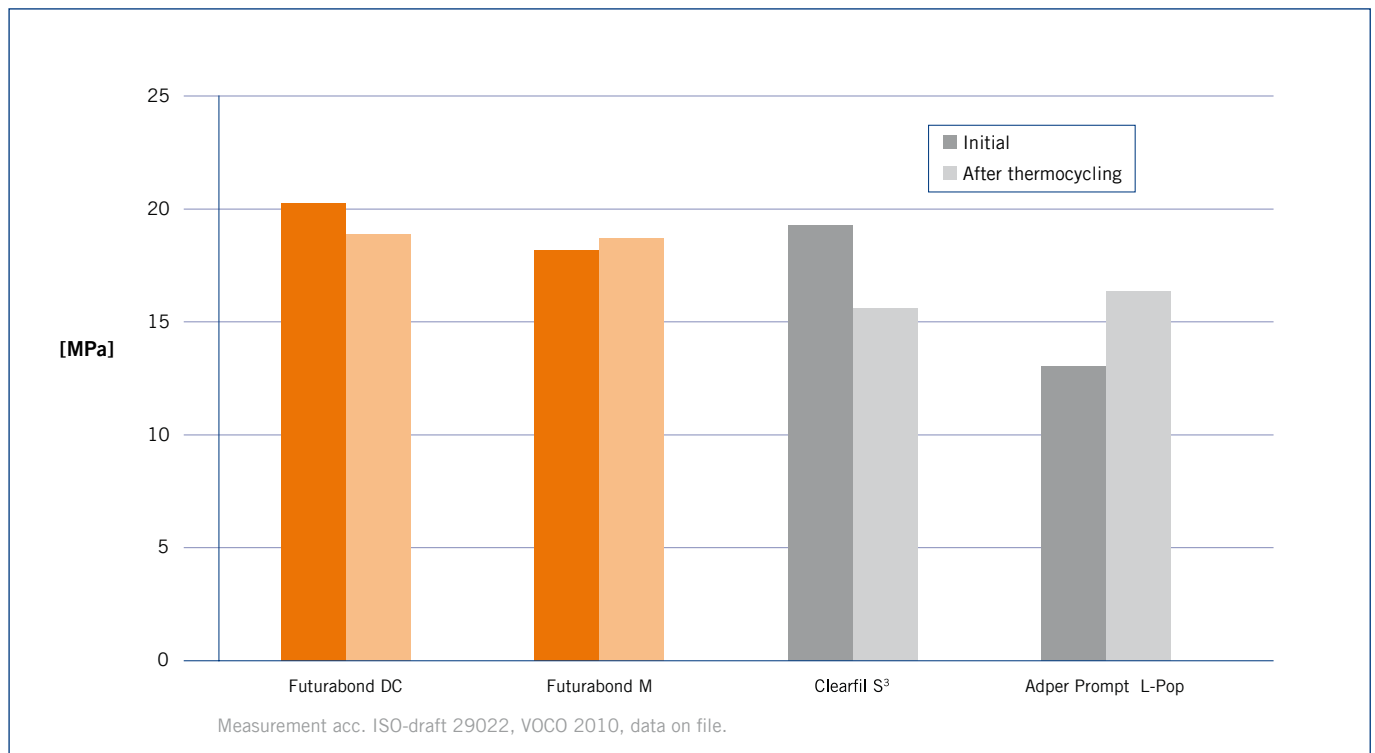
Adhesion values on dentine

Measurement procedure

For this measurement specimens with a diameter of 2.38 mm and a height of 2 mm were prepared and adhesively bonded to human dentine. The dentine surface was roughened with silicium carbide abrasive paper (120 and 400 grit) prior to the bonding procedure. Specimens were subjected to a shear-bond test in an Ultradent device analogous to the ISO-draft standard. A second measurement with equally prepared specimens was performed after thermocycling (3000 cycles, 5°/55°C).^[1]

Results

GrandioSO shows good adhesion values with the bonding materials used, both during the initial stage and after the simulated ageing through thermocycling. The best values were obtained with Futurabond DC and Futurabond M.



Adhesion values [MPa] of GrandioSO on dentine with different bonding materials, initial and after thermocycling.

Literature

[1] ISO-draft 29022.

Summary

Regarding the strength of composites high values are desirable. Measurements of flexural strength both before and after artificial ageing certify that GrandioSO has the highest values in the evaluations presented here. GrandioSO also delivered one of the best values in the determination of fatigue fracture resistance. And concerning compressive strength, GrandioSO even exhibited a higher compressive strength than enamel. Until recently, such high strength could only be achieved with the use of indirect, metal or ceramic restorations. Even in comparison to most amalgam alloys, GrandioSO is ahead with a compressive strength of 439 MPa. Moreover, the high stability under pressure is validated by the high diametral tensile strength. GrandioSO also exhibits an excellent value in the edge strength, a value that is especially important for the restoring of load-bearing cusps in the posterior range. GrandioSO is characterised by an extremely low creep and permanent set and sets new standards in these material properties. GrandioSO showed good adhesion values with all of the tested bondings.

With reference to strength and stability, GrandioSO is not only always found in the top group of the individual disciplines, but it also has an outstanding rank through the sum of these physical properties.

GrandioSO – Surface Optimised

GrandioSO – Physical parameters regarding surface properties

The manufacturer is presented with a special challenge when it comes to the optimisation of the surface of restoratives. A maximum surface hardness and wear resistance is desirable for the durability of a filling. The long-term abrasion can be minimised by high surface hardness; the surface thus remains intact as well as smooth and shiny for longer. A very high wear resistance, however, also represents a certain challenge for the dentist regarding polishing. The hardness, abrasion behaviour and polishability relationships will be discussed in more detail in this chapter.

Surface hardness

The surface hardness indicates to what extent a material provides resistance against loads on a small surface. The smaller the impression left on the surface in such a test, the higher the surface hardness. There are different kinds of hardness, depending on the geometry of the points used in these compression tests: Vickers hardness (pyramid-shaped indenter), Brinell hardness (sphere), Knoop hardness (rhombic diamond point) and Barcol hardness (truncated cone). A surface hardness comparable to enamel is desirable for an occlusal surface with long-term shape stability.

Abrasion

The abrasion describes to what extent abrasive wear of a material takes place on the surface. The so-called ACTA method has been established in dentistry for measuring this parameter. The ACTA abrasion is a test method that was developed by the University of Amsterdam (Academisch Centrum for Tandheelkunde Amsterdam). The long-term abrasive wear from “chewing” solid food particles is simulated in this method. A restorative should exhibit the highest abrasion resistance possible.

Polishing

The finishing is the subsequent step after placement of the filling. This involves removing any excess, carefully contouring and finishing as well as final polishing. Soft composites can be polished in only a few seconds with nearly every polishing system. Such soft materials, however, abrade on their surface relatively fast and the initial gloss is lost soon. One of the development objectives is thus to combine a high surface hardness and wear resistance with good polishability. Furthermore, a rough surface would favour the adhesion of bacteria as well as the uptake of discolourants in the superficial composite layer. With deliberately very hard and resistant materials, such as GrandioSO, special attention should particularly be paid to carefully finishing of the restoration. With modern, nano-hybrid composites, two-step finishing with red and yellow diamonds on medium-speed with water-cooling is recommended. High gloss polishing is subsequently carried out by using slightly abrasive polishing systems.

Evaluations

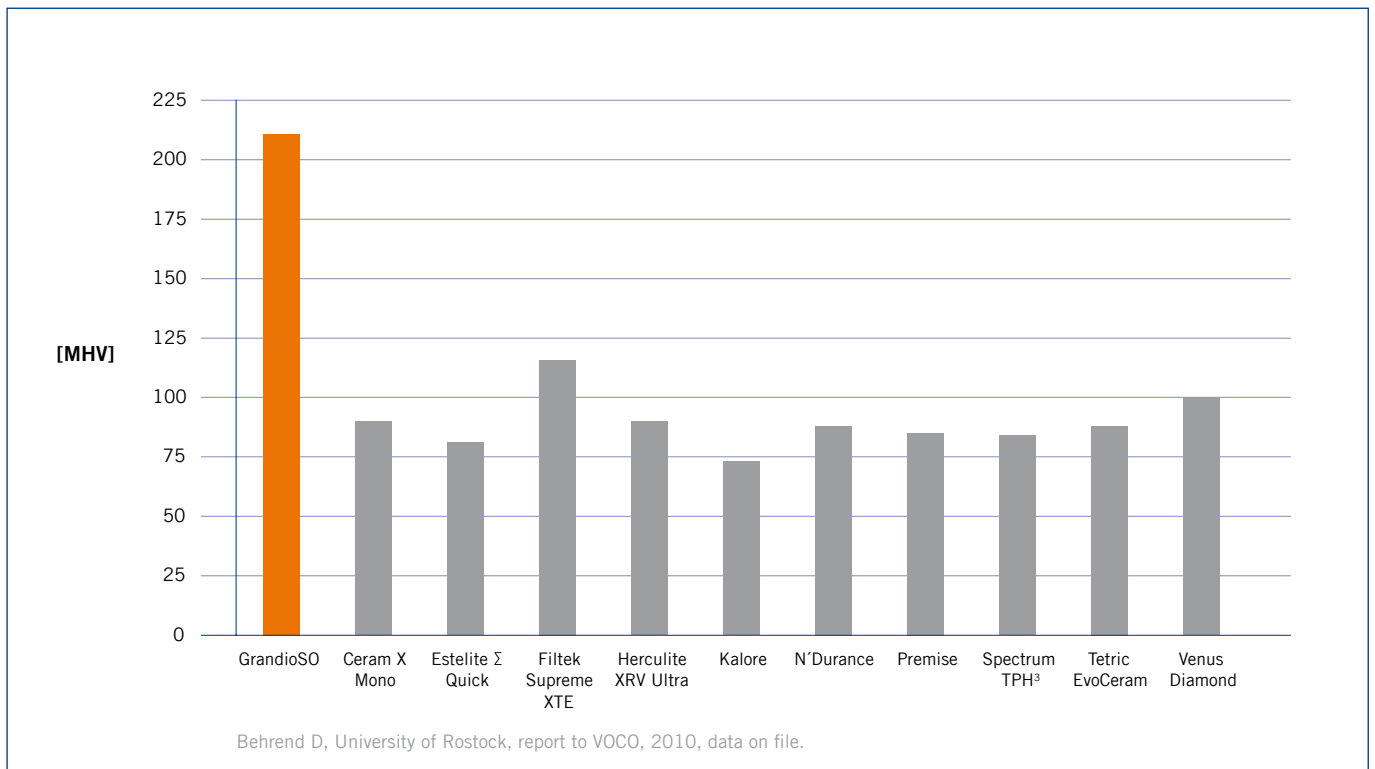
Surface hardness

Measurement procedure

The surface hardness of GrandioSO was determined in a study by the University of Rostock. The micro-hardness (according to Vickers) was measured on light-cured, 2x2 mm test specimens for this purpose.^[1] The surface was initially treated with sandpaper. Subsequently, a standardised diamond prism with a force of 1 N and a penetration speed of 0.2 N/seconds was placed on the test specimens. The diamond was removed again after an exposure time of 5 seconds and the impression remaining in the test specimen was measured. The Micro-Vickers hardness could then be calculated from the dimensions of the impression.

Results

In this test, GrandioSO exhibited, on average, a surface hardness twice as high as all of the other tested materials. The hardness of GrandioSO additionally comes closest to natural enamel (350-450 MHV, green line).^[2] This high value promises long-term resistance to abrasion processes on the surface as well as high shape stability of the occlusal surface.



Surface hardness [MHV] of different composite materials.

Literature

[1] Behrend, 2010.

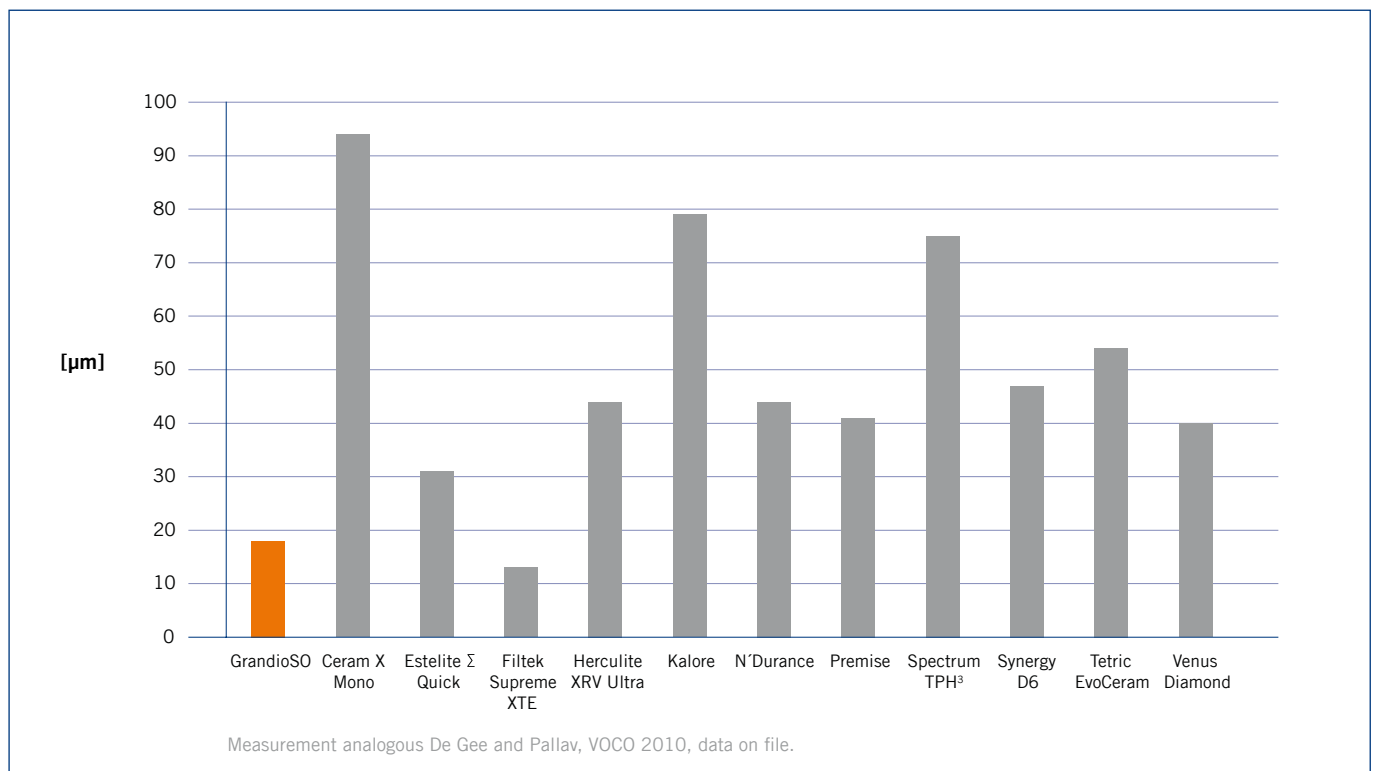
ACTA-abrasion

Measurement procedure

The 3-body ACTA-abrasion was determined according to the method developed at the “Academisch Centrum for Tandheelkunde Amsterdam”.^[1] In this test, the material was applied to a wheel that rotates at 60 rpm. A second wheel made from steel moves in the opposite direction with a pressing force of 15 N. A porridge made from ground rice and ground millet is found between the two wheels. The abrasion of the composite material is measured after 200000 cycles.

Results

Only a minimal amount of abrasion was determined for GrandioSO with 18 µm. Such high resistance to abrasion is a guarantee for long-term intact surfaces as well as lasting gloss of the filling.



Abrasion values [µm] of different composites.

Literature

[1] De Gee und Pallav, 1994.

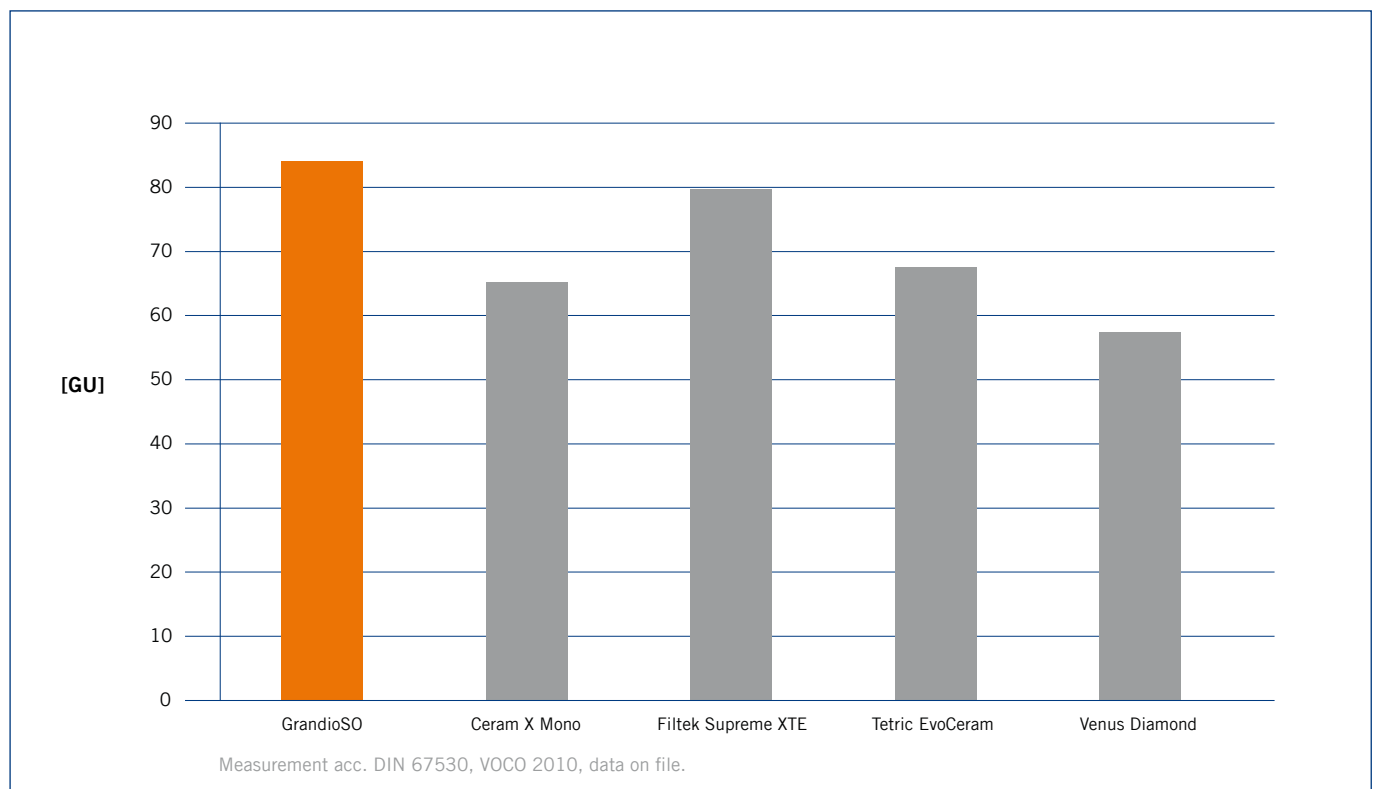
Surface gloss after polishing

Measurement procedure

The surface gloss of the materials was determined on polished test specimens with the assistance of a gloss measuring device in gloss units (GU). Initially, approx. 2 mm high and 15 mm in diameter test specimens were fabricated for preparation. These were subsequently sanded with sandpaper (1000-grit) and then cleaned with iso-propanol. The materials were polished with the Dimanto polisher (VOCO) at approx. 5000 rpm without water-cooling to produce an optimal gloss.^[1]

Despite the very high surface hardness and excellent abrasion resistance of GrandioSO, it is possible to carry out high gloss polishing of the surface. The determined gloss values in this measurement are even higher than the gloss values of the other tested composites.

Results



Values for surface gloss [GU] of the tested composites.

Literature

[1] DIN 67530.

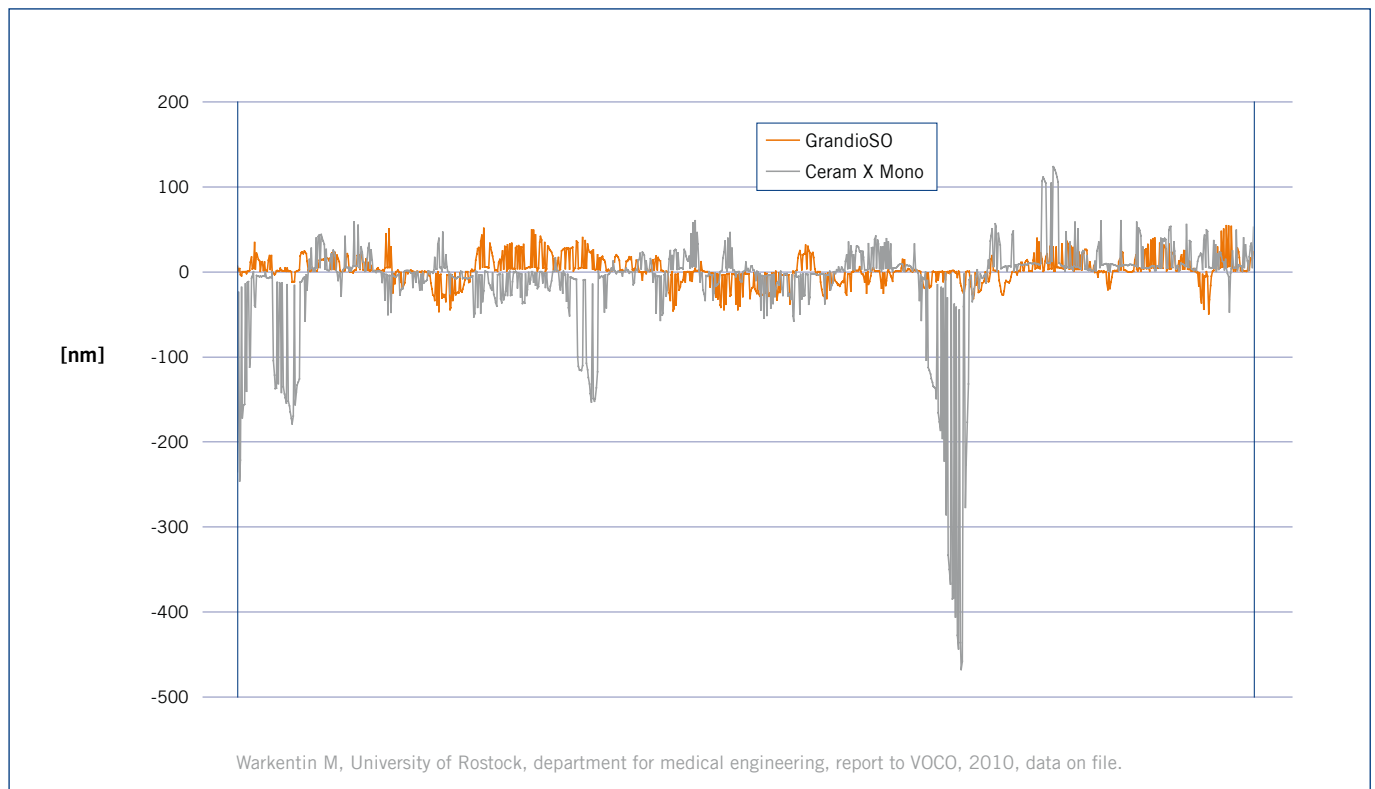
Surface roughness I

Measurement procedure

Atomic force microscopic (AFM) micrographs were taken of polished surfaces of GrandioSO and Ceram X Mono at the University of Rostock.^[1] In this measuring procedure, a needle with a tip consisting of only a few atoms is driven over the surface. The needle bends due to interactions with the atoms on the surface, even if there is no contact between the needle and surface. Conclusions can then be drawn on the topography of the surface from the deflection.

Results

The surface of GrandioSO exhibits significantly fewer irregularities in comparison to Ceram X Mono.



Surface profile [nm] (Atomic Force Topography) of GrandioSO and Ceram X Mono.

Literature

[1] Warkentin, 2010.

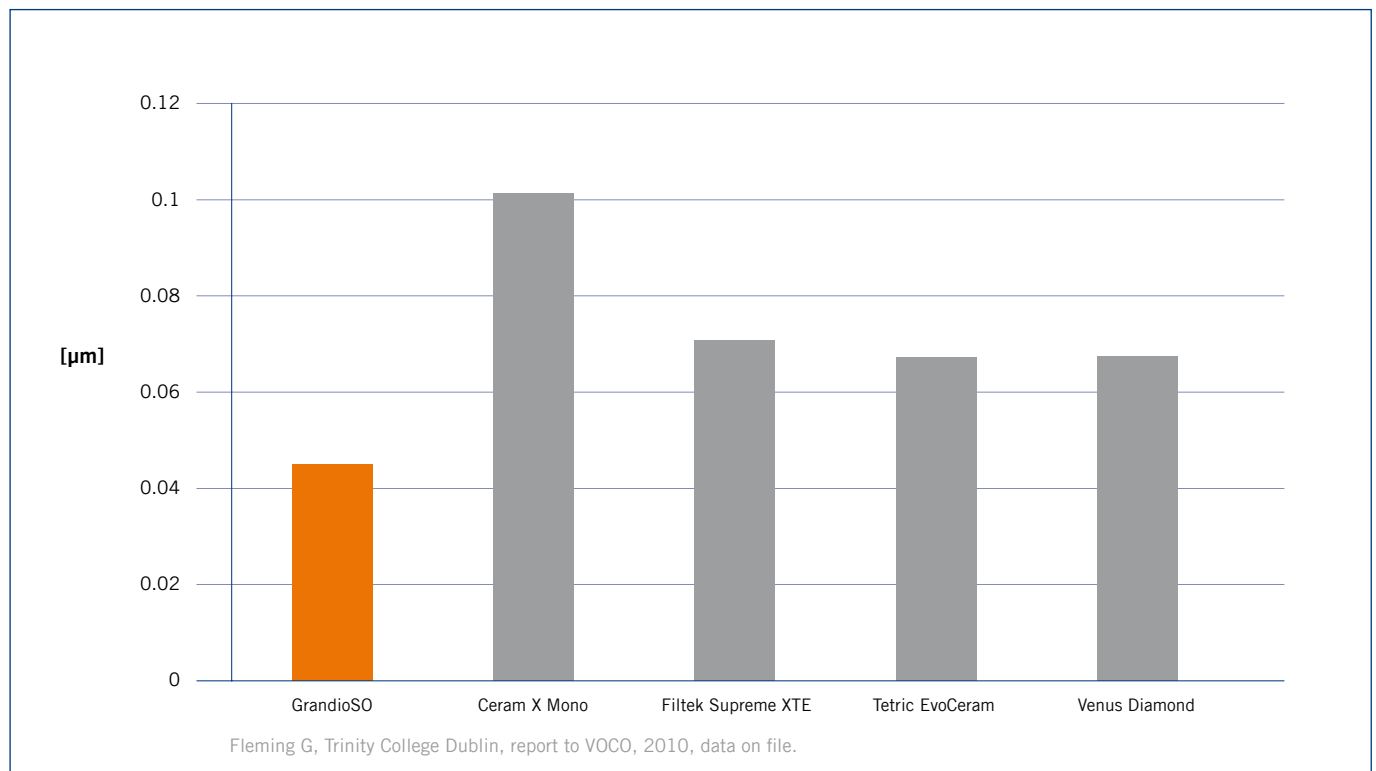
Surface roughness II

Measurement procedure

The test specimens were polished with the Dimanto polishing system after light-curing. The values shown here, which were measured by Fleming et al., were determined in a no-contact procedure by scanning the surface with a chromatic confocal sensor. These sensors can determine distances by the wavelength-dependent exit angles of the light, according to division of the visible light by a lens. The scans were conducted on a 12×12 mm surface and a height determination was conducted every 5 micro-meters, both in x- and y- direction. The 2401 values obtained in this manner have a resolution of 0.0236 nm in the z-direction.^[1]

Results

GrandioSO exhibited the best values in this examination with an arithmetical, average roughness (R_a) of only 0.052 μm . The high surface hardness thus does not prohibit a low surface roughness.



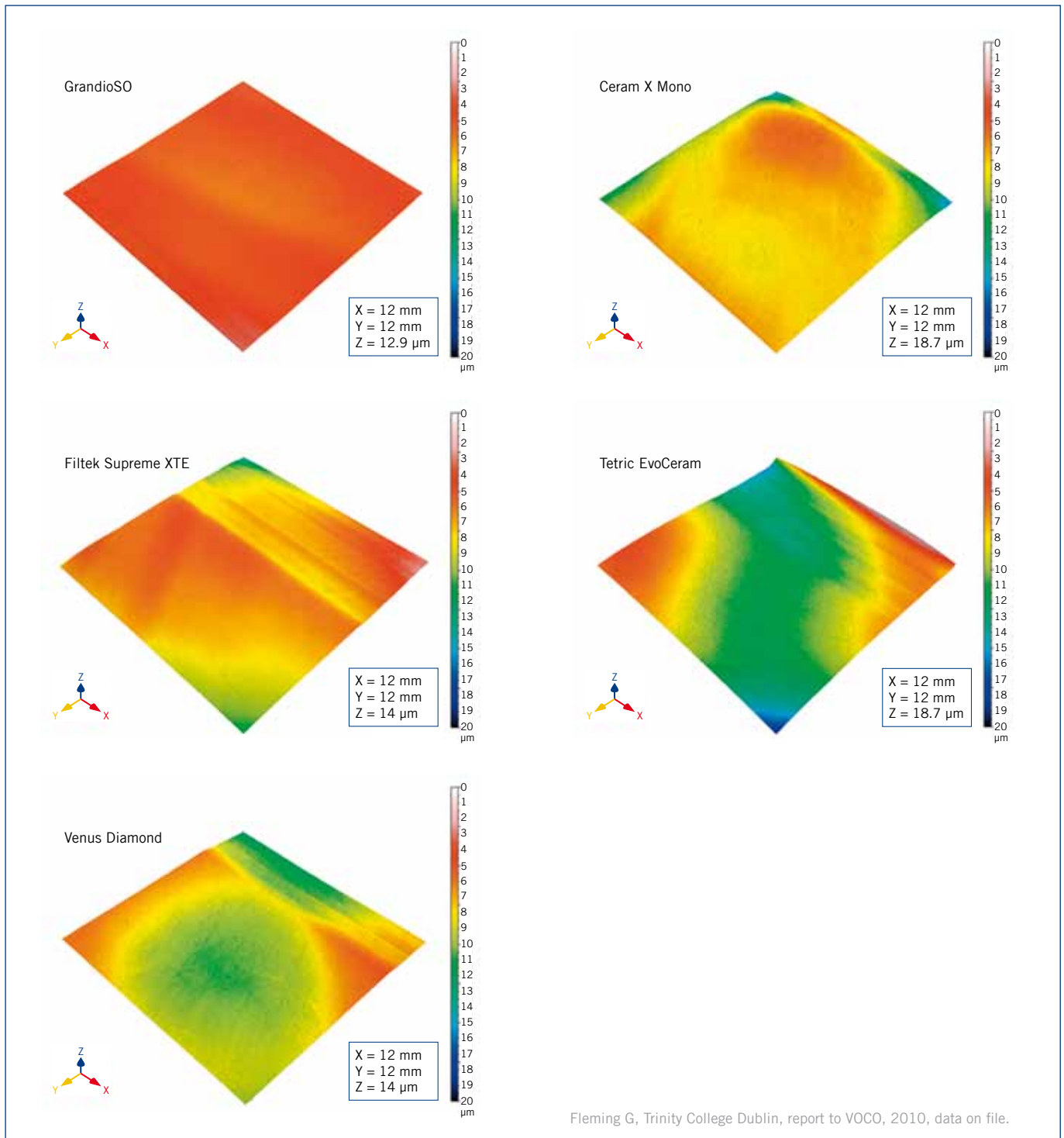
Average roughness (R_a) [μm] of the tested composites.

Literature

[1] Fleming, 2010.

Profilometric micrographs of the examined surfaces were made in the course of the above-shown study by Fleming et al. The colour gradient in these images reflects a change in the height

level within the examined surface. The more monochromatic the image, the lower the changes in the profile.



Profilometric images of the surface of different composites. Lowest changes in the profile of GrandioSO.

Summary

Examinations of the surface show that GrandioSO exhibits a hardness that is twice as high as that of the other composite materials. Moreover, GrandioSO exhibits a high resistance to abrasion processes. These two properties do not inhibit the polishability of GrandioSO. The studies show that a very smooth surface can be achieved at the polishing stage, which likewise leads to excellent values in the gloss measurements.

With GrandioSO, it was possible to develop a composite that optimally combines the requirements of high surface hardness and abrasion resistance with good polishability.

GrandioSO – Solubility Optimised

Physical parameters regarding behaviour in an aqueous environment

Dental restoratives are exposed to the moist-aqueous environment of the oral cavity 24 hours a day. Restoratives are tested to see how they behave in water for this reason, so that the permanent contact to water does not lead to negative consequences, even long-term. Two parameters are meaningful in this respect: The solubility in water as well as the water sorption.

Water solubility

A restorative ideally should be absolutely insoluble in an aqueous environment. Significant dissolution of restorative particles inevitably leads to destabilisation of the restoration. Another important aspect for the determination of the solubility is an inference regarding possible residual monomers. Monomers that are not imbedded in the three-dimensional network during the polymerisation reaction can exit the filling relatively easily. The release of residual monomers, low as it may be, should be minimised as much as possible.

Water absorption

Composites absorb small amounts of water despite their relatively high hydrophobia. This water is stored in the interstices of the polymer. Water storage carries two significant disadvantages. The first disadvantage concerns the volume stability of the filling. When water is absorbed, the material consequently swells. If the water absorption is high, there is a danger that the cavity walls may fracture in the course of time from the pressure of swelling, especially in fillings where only thin cavity walls remain. Enamel cracks may also be a result of swelling of the restorative. The second negative aspect of water absorption is a possible impairment of the aesthetics. Increased water absorption is always accompanied by an increased absorption of coloured substances and thus over time leads to discolouration of the restoration.

Evaluations

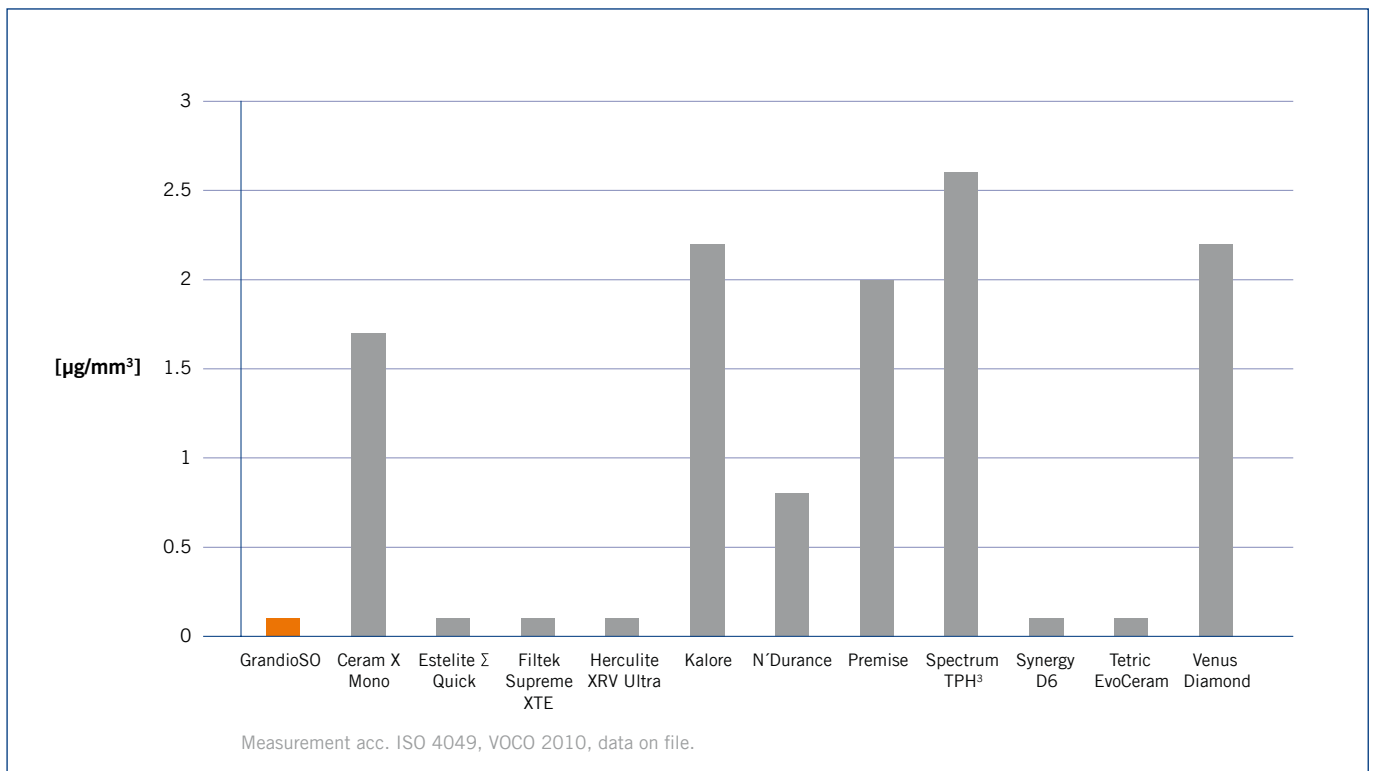
Solubility in water

Measurement procedure

The solubility of GrandioSO in water was determined according to ISO standard 4049.^[1] For this, test specimens with a diameter of 15.0 ± 0.1 mm and a height of 1.0 ± 0.1 mm were light-cured. After determining the initial weight, the test specimens were stored in water at 37 °C for 7 days. The test specimens were subsequently removed, rinsed with water and dabbed until moisture was no longer visible on the surface. The weight was determined again after storage in a vacuum at 37 °C and this weight was compared to the initial weight to determine the resulting water solubility. A water solubility of $\leq 7.5 \mu\text{g}/\text{mm}^3$ is stipulated in the ISO 4049 standard. $0.1 \mu\text{g}/\text{mm}^3$ is the detection limit in this experimental setup.

Results

GrandioSO is characterised by an extremely low solubility in water. A long-term destabilisation from rinsing processes during the retention time of the filling is thus highly unlikely.



Water solubility [$\mu\text{g}/\text{mm}^3$] of different composites.

Literature

[1] ISO 4049, International Organisation for Standardization.

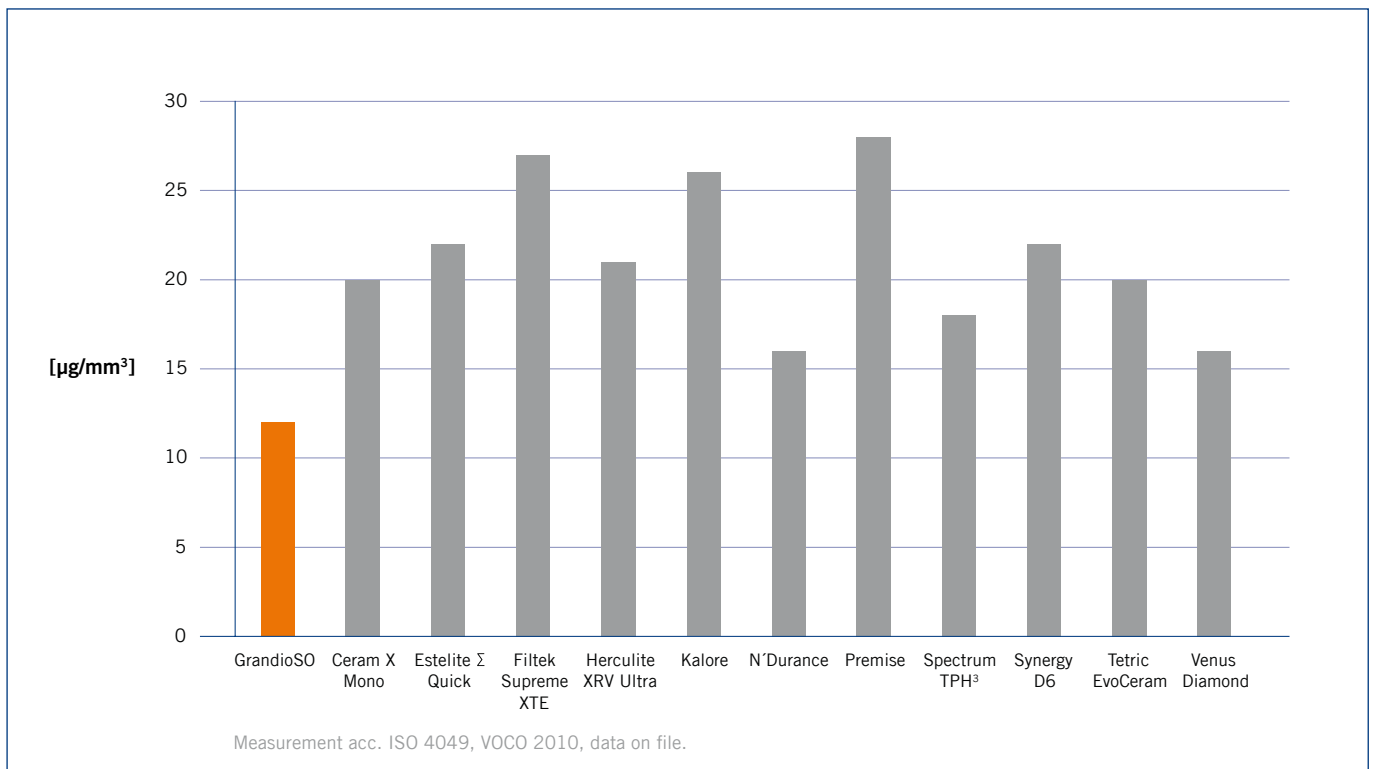
Water absorption

Measurement procedure

The water absorption was determined according to ISO 4049. [1] Test specimens of the tested composites with a diameter 15.0 ± 0.1 mm and a height of 1.0 ± 0.1 mm were light-cured for this. After determining the initial weight, the test specimens were stored in water at 37 °C for 7 days. Afterwards, the test specimens were removed from the water storage, rinsed with water and dabbed until moisture was no longer visible on the surface. The test specimens were waved in the air for 15 s and then weighed 1 minute after removing them from the water. The water absorption results from this value. The ISO 4049 stipulated water absorption of $\leq 40 \mu\text{g}/\text{mm}^3$.

Results

A comparison of the water absorption shows that GrandioSO exhibits the lowest value of the tested composite materials at only $12 \mu\text{g}/\text{mm}^3$. This low amount of water absorption permits the assumption of only minimal swelling behaviour of the filling and a long-term shade stability.



Water sorption [$\mu\text{g}/\text{mm}^3$] of the tested composites.

Literature

[1] ISO 4049, International Organization for Standardization.

Summary

GrandioSO exhibits excellent values in the behaviour in aqueous environment. Solubility as well as absorption are far below the values stipulated by the ISO standards. This behaviour supports long-term intact and aesthetic restorations.

GrandioSO – Speed Optimised Handling properties of GrandioSO

Dental material manufacturers must always keep in mind two aspects when developing new materials. Optimal physical properties of a product are one side of the coin. On the other hand, this product must be easy for the dentist to handle. Among these physical and application-related properties are handling, resistance to ambient light, radiopacity as well as light-curing times of the composite.

Handling properties

As far as the handling of composites is concerned, several important properties are of great interest to the practitioner: sculptability, non-tacky consistency, packability and many more. These parameters were evaluated in a user test in a study by Frankenberger et al. (2010). GrandioSO scored very well in this user survey. The good physical properties could thus be linked to a high amount of user-friendliness.

Resistance to natural light

All light-curing filling composites are caused to polymerise by exposure to blue light. Blue light is, of course, also found in the spectrum of natural light, so that the material slowly begins to harden under daylight. In order to provide the user with a maximum working time and thus stress-free layering, this rather unintentional polymerisation process should proceed very slowly. With a natural light resistance of four and a half minutes, GrandioSO gives the user sufficient working time in any case.

Radiopacity

Good visibility of the restorative in an X-ray image considerably facilitates the diagnosis. At 320 %Al, GrandioSO has very high radiopacity. This permits easy identification of restorations, even in very thin layers.

Light-curing times

The photo-activator in GrandioSO is camphorquinone, which can be activated with all standard light-curing devices. Depending on the opacity of the individual shades and the energy output of the lamps, the following exposure times arise:

LED- or halogen lights with a minimum energy output of 500 mW/cm²

20 s: A1, A2, A3, ^{vc}A3.25, A3.5, A4, B1, B2, B3, C2, D3, BL

40 s: OA1, OA2, OA3.5, ^{vc}A5

LED- or halogen lights with a minimum energy output of 800 mW/cm²

10 s: A1, A2, A3, B1, BL

20 s: ^{vc}A3.25, A3.5, A4, ^{vc}A5, B2, B3, C2, D3

40 s: OA1, OA2, OA3.5

Evaluations

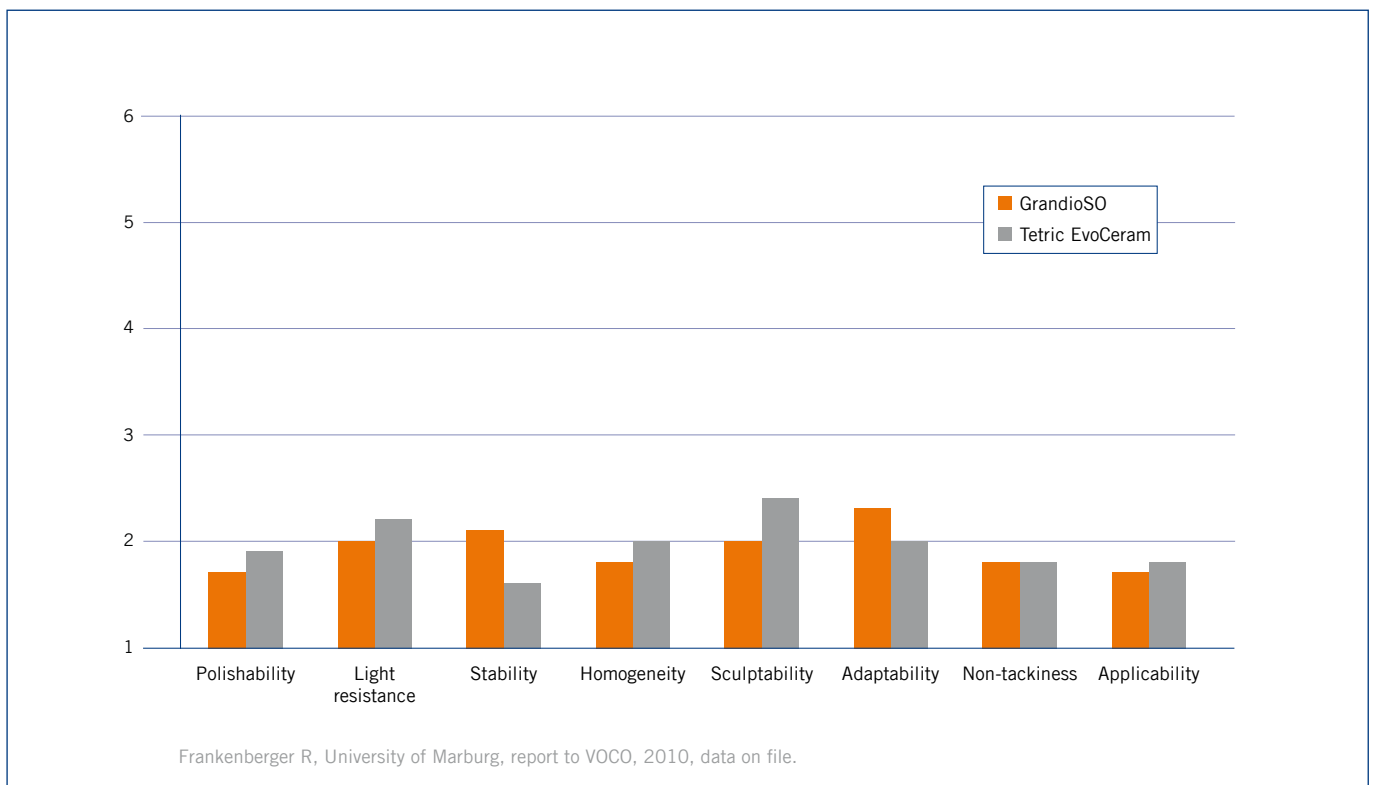
Handling properties

Test procedure

Test dentists were provided with GrandioSO and Tetric Evo Ceram for handling testing in a double-blind user-study by Prof. Frankenberger (University of Marburg). After using these composites, the dentists were asked to provide ratings regarding the different user properties. These were supposed to follow the specified evaluation grid (Very good = 1, Good = 2, Satisfactory = 3, Adequate = 4, Inadequate = 5, Unsatisfactory = 6) for the categories: polishability, light-resistance, stability, homogeneity, sculptability, adaptability, non-tackiness and applicability.^[1]

Results

GrandioSO and Tetric Evo Ceram exhibited nearly matching handling properties. It is interesting here that GrandioSO received a better rated polishability, although it has a considerably higher surface hardness and wear resistance (s. Chapter [GrandioSO – Surface Optimised](#)) in contrast to Tetric EvoCeram.



Handling properties by (German) academic grades.

Literature

[1] Frankenberger, 2010.

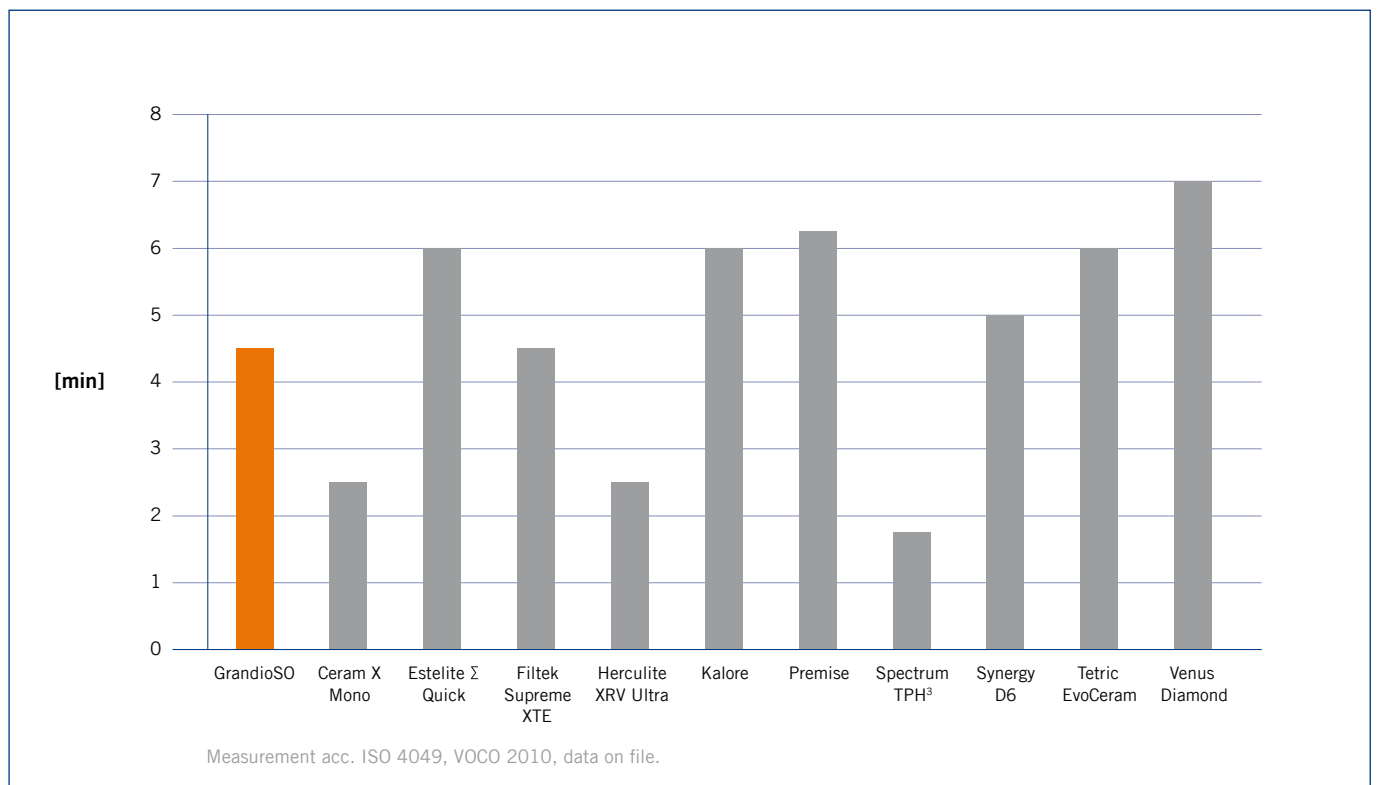
Resistance to ambient light

Test procedure

The natural light or ambient light resistance was determined according to ISO 4049.^[1] Small portions of about 30 mg of the material in the shape of a ball were exposed to a defined ambient light (8000 ± 1000 lux). Each ball was pressed into a thin layer between two glass plates at intervals of 5 seconds. As soon as the material exhibited tears or other inhomogeneities during this procedure, the natural light resistance was deemed to have been exceeded.

Results

A natural light resistance of 4 minutes and 30 seconds permits the user of GrandioSO to carry out a surgery-oriented filling placement, which allows him/her to achieve an optimal result without time pressure.



Resistance to ambient light [min] established for different composite materials.

Literature

[1] ISO 4049, International Organization for Standardization.

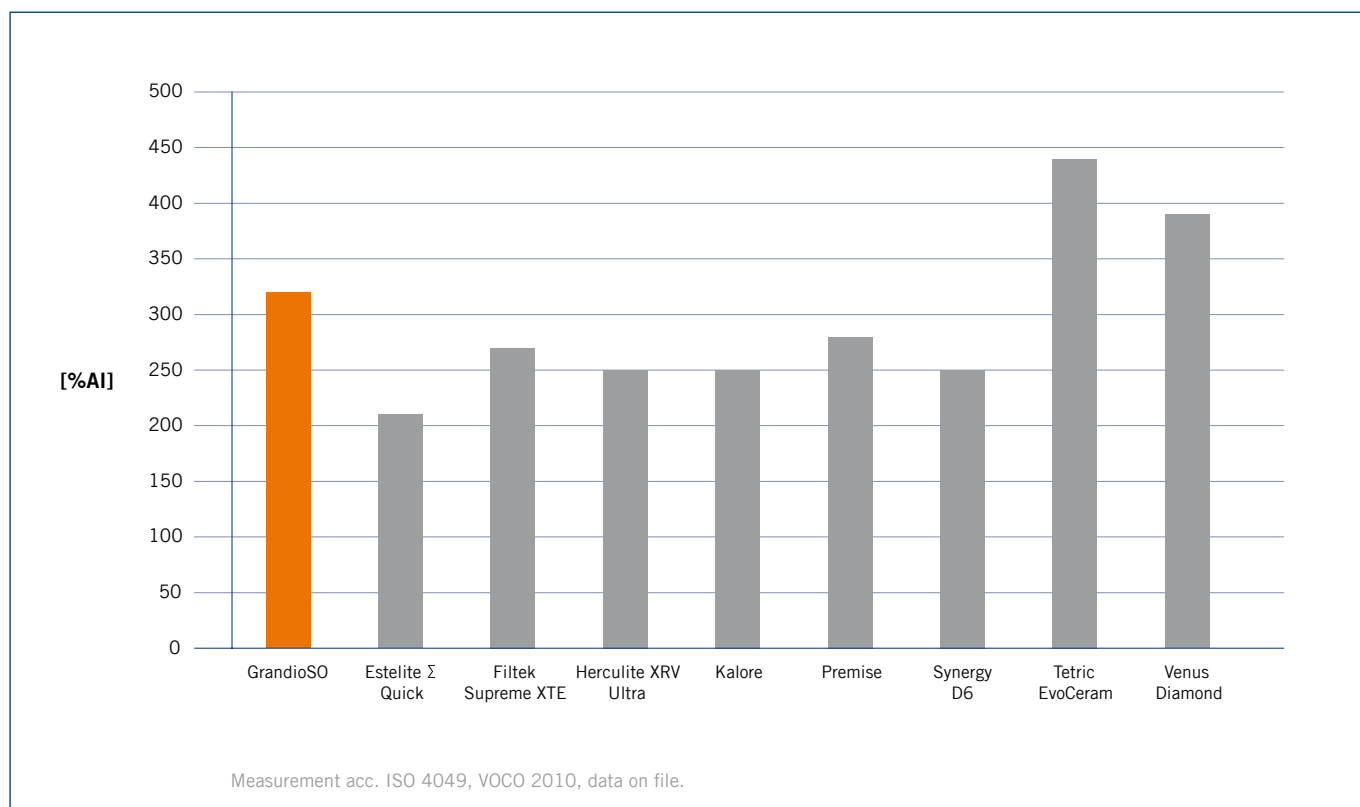
Radiopacity I

Test procedure

To determine the radiopacity, test specimens with a diameter of 15 mm and a height of 2 mm were fabricated. Afterwards, an X-ray of each was taken (7 mA; 60 kV; 0.04 s). A staircase-shaped aluminium body served as reference. For identification, the height of these stairs and the thickness of the test specimens were determined with an accuracy of 0.01 mm. In addition, a determination of the grey values was carried out for both. From these values, the radiopacity was then calculated in aluminium equivalents using linear regression.^[1]

Results

GrandioSO exhibited a radiopacity of 320 %Al. Because of this, excellent visibility in an X-ray image, even if the layers are thin, is guaranteed, which supports the user in his/her initial anamnesis.



Values [%Al] describing the radiopacity of restorative composites.

Literature

[1] ISO 4049, International Organization for Standardization.

Radiopacity II

Clear visibility of fillings in an X-ray image is crucial for the dentist to be able to easily diagnose the clinical situation. To demonstrate the radiopacity of GrandioSO in natural teeth, class II cavities were prepared on the mesial and distal sides of extracted human teeth. After dentine conditioning one side

of the cavity was filled with GrandioSO whereas the other side was treated with a conventional composite. Afterwards digital X-rays were taken (mA= 7, kV= 60, ms= 100).



X-rays of GrandioSO and other composites. Showing differences in radiopacity.

Literature

[1] Braun, 2010

Summary

Test dentists certify that GrandioSO exhibits excellent application characteristics in important areas. Long resistance to ambient light is a prerequisite for application without time pressure. Excellent radiopacity guarantees clear visibility on X-rays and thus facilitates a clear diagnosis.

The outstanding physical properties exhibited by GrandioSO in the studies are accompanied by a high level of application comfort.

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