
Ich bedanke mich bei den unten aufgeführten Kolleginnen und Kollegen für ihre wertvolle Mitarbeit, die sie im vergangenen Jahr geleistet haben.

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Introduction

Clinical studies of composite materials are very time consuming and costly. Furthermore, not every slight material modification can be “tried out” on the patient before continuing with the development of the restorative material. Validated laboratory test methods are necessary to test materials for their clinical suitability within a reasonable length of time. In Part 1 of this review, the general requirements for test methods were discussed and the standardized tests according to ISO were presented. These tests do not cover all of the documentation which exists in material descriptions. Hence, the non-standardized test methods will be addressed in the following; the most common test methods will be presented and compared with the available clinical data. It is clear from the test methods described in the literature that these attempt to imitate the clinical situation. Whether they always succeed or not will be shown in this article.

Materials and Methods

After reviewing the most common test methods and their protocols, a literature search up to February 2010 was performed in PubMed using the following keywords: “composite restoration” AND “modulus of elasticity”, “microhardness”, “volumetric shrinkage”, “expansion”, “optical properties”, “polishability”, “wear”, “handling”.

Physical Tests

Besides the tests included in ISO standard 4049 (ISO 2009), which were discussed in Part 1 of this review article, numerous other physical tests exist for characterizing a composite. Manufacturers of dental products routinely test the modulus of elasticity, Vickers microhardness, shrinkage, and optical properties of a material.
Modulus of Elasticity (Young’s modulus)
The modulus of elasticity (or Young’s modulus), like flexural strength, is a measure of the mechanical stability of a compos-ite. In the test, a bar-shaped specimen fixed at 3 points is loaded to failure in a testing machine at a crosshead speed of 0.5 mm/min. The modulus of elasticity can be read from the slope of the straight-line portion of the resulting stress-strain diagram. It has been shown that the modulus of elasticity is highly dependent on how the material is stored: prolonged water storage or storage in alcohol reduces the modulus of elasticity (Ilie & Hickel 2009). One study reported that composites with a low modulus of elasticity exhibit more marginal fractures, but this has not yet been clinically substantiated conclusively (Rasmussen & Lundin 1995).

Microhardness
The microhardness of a restorative material can be determined with a Vickers or Knoop hardness test. The two methods are similar and differ primarily in terms of the shape of the indenter. The Vickers hardness test was presented in Part 1 of this review. Microhardness depends largely on the filler particles (size, volume percent, weight percent) and the chemical composition (Scougall-Vilchis et al. 2009). Moreover, hardness is influenced by polymerization: the higher the degree of conversion, the greater is the microhardness (Asmussen 1982, Chen et al. 2005). Microhardness is correlated with the modulus of elasticity and viscosity (Li et al. 2009). Studies have shown that the harder a material is, the greater its wear resistance (Mandikos et al. 2001), although beyond a critical value of ca. 500 MPa Vickers hardness, there is no further increase in wear resistance.

Shrinkage
There are essentially three rather elaborate methods of determining shrinkage and shrinkage force: the bonded disk method, the Archimedes test, and the photoelastic method.

Bonded disk method
This method was developed by Watts and Cash (Watts & Cash 1991, Watts & Marouf 2000). The restorative material is inserted into a mold of known height and volume and covered with a thin, flexible glass plate. A detector which can record dimensional changes is placed on the plate. The material is cured through the glass plate, and the dimensional changes are recorded over the desired observation period. The shrinkage force is mathematically calculated.

Archimedes test
In this method, the material densities before and after poly-merization must be recorded. It is crucial to measure the temperature exactly, so that Archimedes’ buoyancy principle can be applied. Using the data, the volumetric shrinkage can be determined with the following formula:

\[ PS = \frac{(\rho_p - \rho_c) \times V}{\rho_c} \times 100 \]  

(PS: polymerization shrinkage in volume percent, \( \rho_p \): density of uncured material, \( \rho_c \): density of cured material).

Volume shrinkage is dependent on the temperature of the material (Lohbauer et al. 2009).

Testing dental composites for volume shrinkage is described by DIN 13907 (German Institute for Standards, 2007).

Photoelastic method
In this test, special photoelastic epoxy resin models are needed. These are obtained from replicas of standard preparations and have a constant volume. In the marginal area of the restora-tion, points of measurement are defined at which the specimen deformation after polymerization of the material is measured under a transmission polariscope. Among other things, the material constant of the photoelastic model material and the deformation data must be included. The salient feature of this method is that the stress distribution across the entire cavity margin is depicted visually, which means the cavity configuration can also be analyzed in terms of stress distribution.

In addition, other tests exist in which extracted teeth are used, for instance, to show how cusps deform during material poly-merization. Still other test methods employ dilatometers or tensiometers to make measurements.

Although shrinkage is seen as the greatest disadvantage of dental composites and all manufacturers work on developing low-shrinkage composites, clinical studies have shown that a composite with ca. 1.5% volume shrinkage does not yield better clinical results than one with ca. 2.5% shrinkage (Manhart et al. 2004). The same applies to the clinical comparison of a composite with 3.1% volume shrinkage vs. a material with 1.7% shrinkage (Vandenbroucke & Lindberg 2009). The shrinkage of current composites ranges from 1.5 to 3 vol% and apparently does not have the critical influence on marginal staining and secondary caries that is generally assumed and has been shown in the laboratory. A clinical study in which mid-sized Class II cavities were bulk filled with only one layer of com-posite and its shrinkage could completely have effect on the margins found that after 3 years, the restorations had no higher rate of marginal staining or secondary caries than did fillings in which the composite was applied in layers (Sarrett et al. 2006). A systematic analysis of the available clinical studies on posterior composite restorations concluded that the frequency of secondary caries and marginal staining did not depend on the type of composite (Brunthaler et al. 2005).

Expansion after Water Sorption
Expansion is determined by measuring the linear change of circular specimens (Ø 20 mm, thickness 1 mm) after water storage using a digital calliper at 4 different points. Despite their hydrophobic character, composite materials absorb water over time, which can not only lead to a degradation of the filler-matrix bonds and thus consequently to increased wear, but it also results in expansion (volume increase). The expansion compensates for and usually exceeds the shrinkage which all composites (without exception) undergo. After about 1 to 3 months of water storage, most composites reach a plateau after which no further expansion occurs. A laboratory study has shown that the deflection of the cusps of three-surface composite restorations that was caused by the shrinkage of the composite resin was compensated by its expansion within 1 month (Versluis et al. 2011). If expansion is too strong, it can lead to cracks in the dental hard tissue, tooth fractures, or even pulpsitis (Van Dijken 2002). Particularly re-restored cavities using large amounts of composite in molars pose an increased risk. Most composites exhibit a linear expansion of less than 0.5%. Composers and even composites that continuously release ions show greater expansion (Watts et al. 2000, Yap et al. 2003). A linear expansion of over 0.8% can be viewed as critical.

Optical Properties
Besides matching a material’s colors to those occurring natu-rrally, other factors such as opacity and transparency are im-
important for esthetics. The transparency of a material describes its ability to transmit light. A material is termed “translucent” if light can pass through it only partially. Transparency is an important parameter for judging how well the material integrates into the existing dental hard tissue. It is determined by irradiating polymerized specimens with light and measuring that portion which is transmitted. Compared to water, the transparency of composites ranges from about 10% to 14%; that of opaque composites is even less (Yu & Lee 2008a).

Fluorescence can also be determined. Using a reflection spectrophotometer, specimens are analyzed against a white and a black background to determine the standard color parameters or coordinates L*a*b* (CIE 1986). Measurements are conducted with and without UV light. Taking the difference between the individual color parameters into consideration, the fluorescence of a material is determined under given different light sources. Aging processes can negatively influence fluorescence (Lee et al. 2005). The comparison of various materials showed that at roughness values between 0.3 and 1 μm, the gloss is almost unchanged, while from 0.3–0.1 μm, the surface gloss increases exponentially (Fig. 1). At a Ra value of approximately 0.1 μm, high gloss is attained. This proves that roughness measurements are suitable for discriminating between rough surfaces, whereas determining the gloss allows distinctions to be made between surfaces that have already been polished smooth. A Ra value of 0.2 μm is seen as the threshold for increased accumulation of oral biofilm (Quirynen et al. 1996). However, this value was determined in vivo on the surface of titanium implants, and it is not certain whether it is also valid for composite surfaces.

When testing a composite with a rubber polishing disk, the pressure with which it is applied is a relevant factor. Most manufacturers of polishing instruments recommend a press-on force of about 200 g (2 N). This force can be checked by pressing the handpiece onto a balance, which shows that the press-on force in clinical situations is usually higher. Doubling the press-on force (400 g, 4 N) led to a considerably worse polish in microhybrid composites such as Tetric EvoCeram (Ivoclar Vivadent). In contrast, the press-on force had no influence on the results of polishing the microfilled composite Heliomolar (Ivoclar Vivadent) (Heintze et al. 2006c).

An important factor is the amount of time required for polishing; considerable differences exist between various composite materials (Fig. 2a, b). On Tetric EvoCeram (Ivoclar Vivadent), polishing 10 s each with Astropol F and Astropol P produces a gloss that is reached on the material Venus (Heraeus Kulzer) only after a further 15 s with Astropol HP (Fig. 2a, b).

### Simulated Aging of the Composite

### Surface Properties – Roughness, Gloss, Discoloration

Due to chemomechanical degradation, the polished surface can become rougher and lose its gloss. In addition, exogenous roughness value Ra is recorded, representing the average roughness value of the profile or surface scan (DIN 2008). To measure gloss, simple devices are used which project a light beam onto a flat surface at a certain angle (e.g., 60°) and measure the percent reflected light; this is compared with a standard (usually mirrored black glass).

The quality of the polish is determined by measuring the surface roughness and surface gloss. Surface roughness is measured using mechanical and optical sensors. As a rule, the mean roughness value Ra is recorded, representing the average roughness value of the profile or surface scan (DIN 2008). To measure gloss, simple devices are used which project a light beam onto a flat surface at a certain angle (e.g., 60°) and measure the percent reflected light; this is compared with a standard (usually mirrored black glass).

### Polishing

The polishing ability of a composite material is easily evaluated by the dentist (Barucci-Pfister & Göhring 2009). Several studies have shown that rough composite surfaces exhibit a higher biofilm accumulation rate than do smooth (de Fucio et al. 2009, Ikeda et al. 2007). The caries-relevant bacterium Streptococcus mutans seems to interact with the surface of composites and increase the roughness still further (Beyth et al. 2008). A clinical study found that the proportion of Streptococcus mutans in interdental plaque is higher with composite restorations than with non-restored teeth (Heintze & Twetman 2002), which partly explains the formation of secondary caries at the gingival floor of proximal fillings.

Rough surfaces also correlate with an increased production of crevicular fluid (van Dijken & Sjöström 1998) and are more prone to rapidly accumulate pigments (Lu et al. 2005). Moreover, a rough surface can be uncomfortable for the patient, as the tongue and oral mucous membranes can detect even a slight roughness (Jones et al. 2004).

Polishability can be easily and reproducibly determined in the lab on standardized specimens (Heintze et al. 2006c). This involves pressing the composite into flat metal molds and polymerizing it. To simulate grinding with abrasive instruments, it has proven useful to employ polishing machines with abrasive paper (320-grit silicon carbide) on the specimen to evenly and reproducibly roughen the surface.

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pigments can penetrate into or accumulate on the restoration.

Gloss stability is most important in the esthetically prominent anterior tooth area, especially for large restorations such as incisal build-ups or direct composite veneers (Dunn 1998). In some countries, particularly in Scandinavia, children and adolescents today have more teeth damaged by trauma than by caries (Glendor 2008). These teeth are almost always restored with composite, so that esthetic parameters like gloss stability are important.

In the laboratory, the aging process is simulated by storing specimens in alcohol, at higher temperatures (e.g., 60 °C), or in different staining solutions, such as red wine, coffee, or safrazin. The criteria evaluated are the degree of degradation and the retention of pigments. However, the clinical relevance of these tests is not clear, since no systematic studies exist.

Simulated tooth-brushing is also used as an aging process, after which the gloss reduction or surface roughness increase can be evaluated. In this test, optimally polished specimens are exposed to tooth-brushing action in a toothpaste solution (Heintze & Forjanic 2005). At certain time intervals, the surface change in terms of gloss and/or roughness is measured (Fig. 3). If the composite specimens are exposed to water only during simulated brushing, almost no loss of gloss occurs. The use of toothpastes with greater abrasiveness (RDA > 100) and particle size (>10 μm) leads to faster gloss loss than do less abrasive toothpastes containing smaller particles (McCabe et al. 2002).

While microhybrid composites dull rapidly, microfilled composites seem less sensitive to this type of aging simulation (Fig. 3) (Heintze & Forjanic 2005).

The press-on force of a toothbrush in vivo averages 3.3 N (van der Weiden et al. 1998). An in vitro brushing duration of one hour corresponds to about 21 months in vivo, if one applies 80 seconds as mean brushing time for the whole dentition.

In wear, friction is an important force which has a greater effect on rough than on smooth surfaces.

As a rule, contemporary composites are wear resistant (Sarrett 2005). Today, wear occurs mainly in the occlusal contact areas, but hardly at all in the contact-free filling areas. Only after longer service times (>4 years) are the anatomical contours reduced to varying degrees depending on the patient. The wear of intracoronal fillings is usually self-limiting thanks to the surrounding dental hard tissue. Depending on the material, intracoronal composites show an average wear after 2 years of 60 to 200 μm (CRA 1996), with wear being highest in the first 12 months. However, when composites are used to make crowns or denture teeth, wear is considerably higher, up to an average of 100 to 200 μm after only one year (CRA 2001, Schmid-Schwap et al. 2009).

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Saxer et al. 1998, and assuming further that the buccal and lingual tooth surfaces are brushed evenly while the toothbrush is reaching two tooth surfaces at the same time, and this happens twice a day.

The data on gloss loss after simulated tooth-brushing and clinical data on gloss reduction are comparable to a certain extent (Heintze et al. 2010, Palanappian et al. 2009).

Wear

Wear is the sum of all material loss in the mouth, whether of natural tooth hard substance or restorations. Various types of wear mechanisms can be distinguished, although they occur in the oral cavity more or less simultaneously (Kunzelmann 1998). When two teeth get into contact during biting or swallowing, for instance, when an enamel antagonist hits a composite filling, this is called two-body abrasive wear or attrition. If there is food between the teeth, or when the toothbrush with dentifrice brushes over the teeth, this is termed three-body wear or abrasion. This is also the case when, for instance, particles of composite are worn off during biting and function as “abrasives”. If not merely microparticles are loosened, but rather larger pieces chip off due to fatigue, this is also a type of wear. Besides these mechanical phenomena, chemical mechanisms exist, such as erosion or corrosion, which are caused by acids in food and drink (e.g., sour or citrus fruit, soft drinks, candies) or by stomach acid in patients suffering from reflux or bulimia (Lussi & Jaeggi 2008). Although acids aggressively attack enamel and dentin, they generally have little effect on restorative materials.

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Material losses of 100–200 μm as measured with precision instruments are clinically barely detectable even by experienced examiners, and much less so by patients (Fig. 4 a,b) (PALLESEN & QVIST 2005). In a comparative study of 3 different composite materials (Tetric, Z100, Charisma), no wear difference was found clinically or subjectively on stone replicas after 6 years (BUSATO ET AL. 2001), although a laboratory examination showed significantly greater wear for Tetric and Z100 than for Charisma (ZANTNER ET AL. 2004).

Wear can be important in terms of esthetics, but it has no consequences for biological structures such as the TMJ or periodontium (BERNHARDT ET AL. 2004, CARLSSON ET AL. 2002, GENCÜ 1996, GESCH ET AL. 2004, JOHN ET AL. 2002, SELIGMAN ET AL. 1988). The risk of elongations or tilting is also slight, as shown in studies of patients with tooth-bounded edentulous spaces (GRAGG ET AL. 2001, SHUGARS ET AL. 2000). Therefore, the question of whether wear measurements are relevant is justified, since the measurement techniques are very elaborate and must be done on models (replicas) (MAIR ET AL. 1996).

At the moment, the most advanced device for measuring wear is based on laser technology and is also used in the CAD/CAM field (MEHL ET AL. 1997). Three-dimensional images of the baseline and follow-up models are superimposed and the software calculates the difference (Fig. 4a, b). This method is utterly dependent on the quality of the impression, and is therefore very technique sensitive.

Furthermore, the selection of subjects and the sample size are important. If predominantly subjects with high masticatory force are examined, high wear will be measured; in general, the masticatory force in men is higher than in women, and in younger than in older subjects (SHINOgAYA ET AL. 2001, YEH ET AL. 2000). Masticatory force, various eating habits, and bruxism are presumably the most important reasons of the great differences in wear. A variability of more than 50% of the mean has been recorded (SÖDERHOLM ET AL. 2001, WILLEMS ET AL. 1993, PALANIAPPAN ET AL. 2010).

To simulate wear, chewing simulators are generally employed, in which antagonists of enamel or a synthetic material (e.g., ceramic) exert a certain load or force on the material being tested. Some methods include an artificial (e.g., PMMA) or natural (e.g., millet, poppy seeds) abrasive to simulate the effect of food (ISO 2001). Other methods use only water. During simulation, it is important that the material be exposed to shear forces in order to test the material’s reaction to fatigue. Depending on the device, this is done with stepper or servo engines (Willytec chewing simulator, SD Mechatronik [KUNZELMANN 1998]), or with electromagnetic actuators and a passive sliding movement via a rubber socket (chewing simulator CoCoM, University of Zurich [KREICI ET AL. 1990]). In the ACTA machine, tests are conducted in a slurry of millet seed shells (DE GEE & PALLAV 1994), and in another machine (OHSU), a mixture of poppy seeds and PMMA is used. Depending on the simulation device, the results show high variability and sometimes, if the same materials are tested, do not yield the same ranking of test materials, which casts doubt on the methodology’s validity (HEINTZE ET AL. 2005, HEINTZE 2006).

In yet another wear test, standardized antagonists of IPS Empress are used which possess a shape and curvature similar to that of the palatal cusps of maxillary first molars. With a load of 5 kg, the antagonists 120,000 times slide 0.7 mm over the flat composite specimens. Willytec is the chewing simulator used (s.a.). The vertical material loss on the wear facet is measured with laser technology on stone replicas. These results are comparable to those obtained with an optical sensor which takes measurements directly on the specimen or those yielded by mechanical scanning (profilometry) (HEINTZE ET AL. 2006a). The wear generated by Empress antagonists on composite is similar to that from enamel antagonists (HEINTZE ET AL. 2006b). It has been shown that the smaller the fillers, the more fillers per unit volume, and the greater the surface hardness and fracture toughness, the lower the wear is (HEINTZE ET AL. 2007).

It is difficult to correlate wear simulation with the in-vivo situation. For one method, it is claimed that 1.2 million cycles in the simulator corresponds to a clinical service period of 5 years (KREICI & LUTZ 1990), and for another, 100,000 cycles are reported to equal 3.6 months in vivo (BÄRKMIEER ET AL. 2004). These correlations are not based on comprehensive longitudinal studies, and the measurements were linearly extrapolated, although wear increase has been proven to progress non-linearly (SÖDERHOLM ET AL. 2001).

Handling Properties

The handling properties of composite materials have not been scientifically examined to any great extent, and this test is highly subjective and dependent on the preferences of individual operators.

As a rule, dental companies conduct “market tests” with potential users prior to a product’s commercial release. During the development of a new composite material (Tetric EvoCeram), handling tests were conducted in which a total of 70 dentists participated. In addition to two versions of the new composite, the participants also tested an older, established product (Tetric Ceram) – once with the original labels and once with neutral packaging. The parameters evaluated were sculpting properties, stability, packability, adherence to the instrument,
No validated measuring methods exist for any of the parameters mentioned above that could objectify the subjective assessments. For the parameter “consistency”, there are different testing devices, such as the penetrometer, which drops a thin metal needle with a load of 50 g onto unpolymerized composite and measures the needle’s penetration depth (Mutlu et al. 1992). The penetration depth ranges from 3 to 8 mm, depending on the consistency/viscosity of the composite. Another method (Rheometer) “excites” the composite with oscillating forces and records the viscous and elastic phases (Lee et al. 2007). Although it is possible to broadly define different composite classes with these devices, they usually do not agree with dentists’ subjective assessments.

Conclusion

Before starting clinical testing, laboratory tests are useful and necessary to estimate risks associated with restorative materials in terms of function, esthetics, and longevity. To be able to adequately assess these functions with sufficient prognostic reliability, a series of relatively simple test methods exists, which in part also have a clinical correlate, e.g., the test of expansion. Other tests are less important, such as surface hardness or shrinkage, since for parameters like these, almost all materials fall within a relatively narrow range, which does not lead to clinically detectable differences.

Wear can be tested with widely differing techniques, but to date there is no recognized standard test protocol. Although it is possible with great effort to measure occlusal wear, it is often not subjectively perceptible; and even if it is noticeable, wear is at most only esthetically important.

The dentist him- or herself can easily test certain parameters, such as polishability and esthetic properties. If the practitioner has obtained satisfactory results over a longer time period with a certain restorative material, he or she should only switch to a new material if clinical studies clearly show that the new material provides better results compared to a standard material. Certain laboratory simulations should be more carefully examined in terms of clinical correlation and accordingly adapted. Only then can these test methods be considered valid for predicting clinical outcome (Tab. I).

Résumé

La première partie de cet aperçu relatait les dispositions d’autorisation et tests de laboratoire aux normes ISO. Dans la deuxième partie, les tests non standardisés utilisés pour évaluer les matériaux composites sont présentés et discutés. Il s’agit principalement de tests mesurant les propriétés physiques telles que l’analyse de la brillance de surface et son altération dans le temps, ainsi que les simulations de vieillissement des matériaux dentaires. L’importance de chaque test de laboratoire et sa corrélation clinique sont évaluées. Comme ces tests ne sont pas réglementés par une standardisation ISO, des différences dans les protocoles entre laboratoires existent et peuvent influencer les résultats ainsi que les conclusions pour leur usage clinique. Les résultats individuels pour un produit donné d’un test particulier ne devraient donc pas être directement comparés entre instituts, mais le classement (ranking) des produits testés devrait être le même entre les protocoles. Les tests montrent une pertinence clinique qui sont les mesures du module d’élasticité, de l’expansion après absorption d’eau et le polissage. Les autres tests tels que la duréité de surface ou la contraction de polymérisation se retrouvent dans une bande de va-
leurs étroites sans répercussions cliniques mesurables. L’usure occlusale souvent mesurée avec des moyens importants n’est que peu perceptible. Une propriété difficile à standardiser est la manipulation du matériau (handling), car son évaluation est hautement dépendante de la préférence de l’opérateur.

References


Beyth N, Bahri R, Matalon S, Domb A J, Weiss E I: Wear No No Slight

Beyth N, Bahri R, Matalon S, Domb A J, Weiss E I: Tooth-brushing simulation No No Questionable (Yes)

Boiling in staining solutions No No Questionable


