Special feature

Ivocerin® – a milestone in composite technology
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The 2-mm increment technique has been the standard application method for direct dental composites for many years. Over time, the properties of resin composites have been consistently improved and the bond strength of dental adhesives optimized. In contrast, the light initiators used in composite filling materials and the light-emitting efficiency of the photo-polymerization process remained unchanged for many years. However, further developments have now also been achieved in this regard. In a series of scientific articles, we present the fundamentals of photo-polymerization and information on the new photoinitiator Ivocerin (Ivoclar Vivadent). We describe the development, working mechanism and application of a new class of photoinitiators for the visible light spectrum and light polymerization in general. Ivocerin has enabled us to develop a new bulk fill material (Tetric EvoCeram Bulk Fill, Ivoclar Vivadent) that can be applied and cured in 4-mm bulk increments.

The new Ivocerin photoinitiator is characterized by high quantum efficiency, high absorption capacity and very good bleaching properties.

Further gradual development
After obtaining very promising fundamental research data, we proceeded to optimize the synthesis of Ivocerin in order to reproduce the molecule in large amounts and at a high level of purity. At the same time, the first successes regarding a 4-mm depth of cure were attained with experimental composites. Furthermore, in vitro studies showed excellent results in terms of marginal seal, retention and esthetics. The results were comparable to those of Tetric EvoCeram placed in 2-mm increments. Ultimately, Tetric EvoCeram Bulk Fill was produced on the basis of Tetric EvoCeram, with the addition of Ivocerin and other photoinitiators. The monomer-filler system was also optimized to enhance the esthetics of the material, to reduce shrinkage stress and increase depth of cure. Three technical developments patented by Ivoclar Vivadent have been incorporated into Tetric EvoCeram Bulk Fill: Ivocerin as a photoinitiator (polymerization booster), a light sensitivity filter and a filler technology (shrinkage stress reliever).

Interesting alternative
Ivocerin represents a very interesting alternative to the established photoinitiators camphorquinone-amine, phenyl propandione and acyl phosphate oxide (Lucirin TPO). Ivocerin is capable of absorbing light at a higher wavelength range than acyl phosphate oxide, and can therefore be activated by all commercially available (halogen, LED) polymerization lights (Fig. 1). In contrast to the camphorquinone-amine initiators, Ivocerin can be used to produce amine-free composite resins, which are colour-stable under artificial sunlight and do not interact with acid monomers. For Tetric EvoCeram Bulk Fill however, the combination of Ivocerin plus camphorquinone-amine has proven to be most successful at ensuring an excellent cure in increments of 4 mm (Figs 2 to 5 show a clinical case).

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ight-curing filling materials have been on the market since the 1970s. These materials are composed of monomers and fillers and they additionally contain initiators, stabilizers and additives which are dissolved in the monomer. As the polymerization reaction can be initiated “on demand”, these materials give users sufficient time to apply them in the cavity.

Since light transmission through composite material is only limited, a cavity usually needs to be filled in several increments, each of which is cured separately. Normally, the thickness of each increment must not exceed 2 mm, as complete curing cannot be ensured with thicker layers. In order to increase the admissible maximum layer thickness, all the factors that influence the depth of cure need to be taken into account, including translucency, shading, and the initiators and their concentration, as well as exposure time and light intensity. An important factor in this context is the photoinitiator used. In the case of Tetric EcoCeram Bulk Fill, for example, the photoinitiator Ivocerin developed by Ivoclar Vivadent in cooperation with the Vienna University of Technology has made a major contribution to increasing the admissible layer thickness without detrimentally affecting the optical properties of the composite, such as translucency and shade.

The chemistry of Ivocerin

Our search for an alternative visible light photoinitiator was crowned with success: in cooperation with Prof. R. Liska of the Vienna University of Technology we succeeded in developing tailor-made visible light photoinitiators based on germanium compounds. Even though it was known from the literature that radicals are formed during light-induced cleavage of e.g. organic germanium compounds [1], the potential of these compounds as photoinitiators for the visible light spectrum had not yet been fully recognized when we started our work in this field. We were able to show for the first time that germanium compounds such as benzoyltrimethylgermane (Ge-1) or dibenzoylethylgermane (Ge-2, Fig. 1) represent very efficient visible light photoinitiators for methacrylate resins [2, 3]. Compared to camphorquinone (CQ), Ge-1 and Ge-2 demonstrate much more intensive absorption in the visible region. The quantum efficiency of the light-induced cleavage for Ge-2 was determined to be 0.85, whereas the quantum efficiency for camphorquinone-amine photoinitiators is below 0.10 and thus significantly lower. Moreover, quick decolouration of Ge-1 and Ge-2 was observed upon exposure to light. An analysis of dental composites containing Ge-1 or Ge-2 as photoinitiators [3, 4] also showed that they offer considerable advantages compared to camphorquinone amine-based materials. Apart from demonstrating quicker curing and excellent bleaching behaviour, these materials require a much lower concentration of the photoinitiator to achieve comparable mechanical properties. Similar to camphorquinone amine-based composite resins, Ge-1 or Ge-2-based materials also show high storage stability.

In cooperation with Prof. G. Gerscheidt and Prof. R. Saf of the Graz University of Technology as well as Prof. R. Liska of the Vienna University of Technology, the mechanism of photolysis in diacylgermanes exemplified by Ge-2 was studied using state-of-the-art methods [5]. In the study, light-induced cleavage of Ge-2 was investigated under various conditions.
The examinations conducted confirmed that dibenzoyldiethylgermane Ge-2 forms benzoyl (B·) and germyl (G·) radicals produced by photolytic fission are the radicals that initiate the polymerization reaction. Based on the results of these mechanistic investigations and the evaluation of different synthesis methods and structural variations of germanium compounds, bis-(4-methoxybenzoyl)diethylgermane Ge-3 was selected as the optimum photoinitiator and protected by a patent under the name of Ivocerin (Fig. 3). We were able to efficiently synthesize Ge-3 (λmax = 408 nm) in two stages. It showed the strongest absorption in the visible region [6]. Ge-3 is a solid substance which is not soluble in water and has an intensive yellow colour. Its melting point is at approx. 50 °C. The synthesis of Ge-3 is started by metallating protected 4-methoxybenzaldehyde using n-butyl lithium, which is then followed by a coupling reaction with dichlorodiethylgermane. In a second stage, the protecting group is split off (Fig. 3). The resulting Ivocerin is of high purity (>96% when determined by High Performance Liquid Chromatography - HPLC).

Apart from chemical characterization and application tests, toxicological investigations [7] were conducted with Ivocerin and comprehensive patent protection measures were initiated.

Moreover, two mutagenicity tests were carried out. Neither the Ames test (in vitro) [8] nor the Mouse Mircronucleus Assay (in vivo) [9] revealed any mutagenic effects of Ivocerin.

We were able to obtain comprehensive patent protection for the germanium compounds. The US and EP patents were granted in 2009 [10].

Compared to conventional camphorquinone amine-based photoinitiator systems, the novel germanium photoinitiator Ivocerin is characterized by intensive absorption of light in the visible region and high photoreactivity. Furthermore, it imparts an excellent bleaching behaviour to composite resins. The radicals required to initiate the polymerization reaction are created by light-induced cleavage of Ivocerin. Additional co-initiators or accelerators are not required. The quantum efficiency of radical formation in conjunction with Ivocerin is many times higher than that in conjunction with camphorquinone.

Against this background, Tetric EvoCeram Bulk Fill takes an exceptional position among the currently available composite resins. Apart from providing a reliable four-milimetre cure when irradiated for ten seconds at a light intensity of more than 1,000 milliwatt per square centimetre, Ivocerin allows the opacity of the composite resin to be established at a slightly higher level compared with other bulk-fill materials. The translucency of the material is such that it ensures a seamless blend with the surrounding tooth structure (enamel in particular) due to the composite’s optimized optical properties in combination with a favourable refractive index.

Prof. Dr Norbert Moszner, Schaan (Liechtenstein)

The full version of this article is available in the R&D Report, No 19, published by Ivoclar Vivadent AG in July 2013.

L I T E R A T U R E


Fig. 4: Tetric EvoCeram Bulk Fill is the first material containing Ivocerin. Figures: Ivoclar Vivadent
The photoinitiators Ivocerin and camphorquinone in comparison

Dr Peter Burtscher talks about the particular properties of the photoinitiators used in dentistry

All customary composite filling materials are polymerized with blue light. Therefore, the light-absorbing initiators in the composites have an inherent yellow colour, which is the complimentary colour to blue light (Figs 1 to 3). When composites are cured, the colour of the initiator largely disappears. However, a slight yellowish tinge always remains. As a result, absolutely white restorations cannot be created with materials that cure with blue light. Nevertheless, the light tinge is accepted by dental practitioners, since natural teeth also have a yellow hue.

Figures 1 to 3 show the initiators in their pure form. Camphorquinone (CQ) and Ivocerin in particular, exhibit a very strong yellow colour. Figure 4 compares the absorption spectra of conventional initiators with the spectrum of Ivocerin. Of particular note is the strong absorption of Ivocerin in comparison to that of camphorquinone despite the fact that its concentration is considerably lower. Acyl phosphine oxide, for example, Lucirin TPO, predominantly absorbs light in the UV range. Hence its inherent yellowish colour is very light, as shown in Figure 2.

Investigations with Ivocerin

Extensive studies were undertaken to examine the suitability of Ivocerin in dental composite resins. The aim of these studies was to establish at which concentration Ivocerin would achieve the same curing results as camphorquinone. Generally, a concentration of 0.3 wt% camphorquinone is added to the monomer. At lower concentrations, reactivity declines. At higher concentrations, the depth of cure decreases, because more blue light is absorbed by the inherent colour of this initiator.

Camphorquinone cannot be used on its own. It always has to be used together with a co-initiator, usually in the form of a tertiary aromatic amine. In a comparative investigation of the reactivity of Ivocerin and a standard monomer containing 0.3 wt% camphorquinone and 0.6 wt% amine, monomers containing 0.1 to 0.4 wt% Ivocerin were mixed and tested in a Tetric-like formulation.

Table 1 shows that 0.2 percent Ivocerin in the monomer achieves comparable results to the conventional amount of camphorquinone and amine. Higher concentrations of Ivocerin increase the strength of the composite.

Influence of the concentration on the depth of cure

Similarly to the flexural strength findings, Table 2 shows that a concentration of 0.2 wt% Ivocerin also achieves good results in terms of the depth of cure, that is, the same as the standard concentration of camphorquinone and amine. A concentration of 0.4 wt% Ivocerin in the monomer increases the depth of cure significantly.

Combination with other light-activated initiators

It was interesting to find out whether or not Ivocerin offers improved depth of cure in...
combination with other initiators. The ideal choice was to combine it with camphorquinone and amine. In this case, the amine concentration had to be twice as high as that of camphorquinone.

The determination of the depth of cure is a quick method for examining the reactivity of a composite. Vickers hardness tests, however, provide more meaningful results. The composite material is polymerized in the recommended layer thickness and Vickers hardness values are established at the top and bottom of the sample. According to a study by David Watts (University of Manchester), the material is adequately cured when the hardness established at the bottom of the sample corresponds to at least 80% of that measured at the surface [Watts D, Amer O, Come E. Characteristics of visible light-activated composite systems. Br Dent J 156 (1984) 209-215]. Therefore, the influence of the initiator system on the depth of cure (DOC, irradiation with Bluephase G1 in HP mode for 10 seconds) and the Vickers hardness (same irradiation) was evaluated.

The results show that the depth of cure and the hardness of 4-mm increments significantly improve in the cases where Ivocerin is used together with camphorquinone. However, it is also evident that higher concentrations of Ivocerin do not lead to further increases in the curing performance (Table 3).

**Summary**

The different investigations on Ivocerin show that this initiator can be used successfully as an alternative to camphorquinone-amine. Very good product properties are achieved when Ivocerin is used in combination with camphorquinone-amine. Excellent depth of cure is attained with 4-mm increments. Therefore, this initiator combination was used for the first time in the innovative Tetric EvoCeram Bulk Fill composite.

The fundamental research on Ivocerin has been incorporated into the development of Tetric EvoCeram Bulk Fill. Given the various influences of Ivocerin on the physical properties of a composite resin, the concentration of this initiator was deliberately kept low in this product. As a consequence, the colour of the filling material before polymerization is only moderately influenced and the shrinkage stress is reduced to a minimum. The resulting composite resin is available in three popular basic shades to cover the esthetic demands of posterior teeth. Furthermore, the filling material can be placed in 4-mm thick increments, which are polymerized in 10 seconds with the Bluephase Style curing light (1,100 milliwatts per square centimetre – mW/cm²).

**Table 1:** Comparison of the flexural strength and modulus of elasticity in relation to the different concentrations of Ivocerin and the standard initiator system

<table>
<thead>
<tr>
<th>Initiator in monomer</th>
<th>Flexural strength (MPa)</th>
<th>Modulus of elasticity (MPa)</th>
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<tr>
<td>0.10% Ivocerin</td>
<td>124 ± 8</td>
<td>7200 ± 300</td>
</tr>
<tr>
<td>0.15% Ivocerin</td>
<td>124 ± 14</td>
<td>8500 ± 200</td>
</tr>
<tr>
<td>0.20% Ivocerin</td>
<td>135 ± 9</td>
<td>9400 ± 600</td>
</tr>
<tr>
<td>0.40% Ivocerin</td>
<td>140 ± 15</td>
<td>10300 ± 300</td>
</tr>
<tr>
<td>0.30% CQ and 0.60% amine</td>
<td>138 ± 10</td>
<td>9200 ± 600</td>
</tr>
</tbody>
</table>

**Table 2:** Comparison of the depth of cure in relation to the different concentrations of Ivocerin and the standard initiator system

<table>
<thead>
<tr>
<th>Initiator in monomer</th>
<th>Depth of cure (mm)</th>
</tr>
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<tbody>
<tr>
<td>0.2% Ivocerin</td>
<td>4.7</td>
</tr>
<tr>
<td>0.4% Ivocerin</td>
<td>5.1</td>
</tr>
<tr>
<td>0.3% CQ / 0.6% amine</td>
<td>4.7</td>
</tr>
</tbody>
</table>

**Table 3:** Depth of cure (DOC) and Vickers hardness of composites containing various concentrations of the initiator

<table>
<thead>
<tr>
<th>Initiator concentration in monomer</th>
<th>DOC (mm)</th>
<th>Vickers hardness at surface (N/mm²)</th>
<th>Vickers hardness at bottom, 4 mm Value (N/mm²)</th>
<th>% of the surface hardness</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.2% Ivocerin</td>
<td>4.7</td>
<td>489</td>
<td>333</td>
<td>68</td>
</tr>
<tr>
<td>0.2% / 0.1% CQ</td>
<td>4.9</td>
<td>511</td>
<td>378</td>
<td>74</td>
</tr>
<tr>
<td>0.2% Ivocerin / 0.2% CQ</td>
<td>5.1</td>
<td>524</td>
<td>414</td>
<td>79</td>
</tr>
<tr>
<td>0.4% Ivocerin</td>
<td>5.1</td>
<td>536</td>
<td>437</td>
<td>82</td>
</tr>
<tr>
<td>0.4% Ivocerin / 0.3% CQ</td>
<td>5.6</td>
<td>538</td>
<td>462</td>
<td>86</td>
</tr>
<tr>
<td>0.6% Ivocerin / 0.3% CQ</td>
<td>5.6</td>
<td>546</td>
<td>465</td>
<td>85</td>
</tr>
</tbody>
</table>
Since its introduction almost 10 years ago, Tetric EvoCeram has been producing excellent clinical results. The composite filling material meets high standards with regard to its longevity and esthetics. Furthermore, it shows very good shrinkage behaviour and surface characteristics (polishing properties and wear resistance).

Since the new bulk-fill material is intended for use without a capping layer, its surface properties have to meet the same requirements as those of Tetric EvoCeram. Alongside having to satisfy these typical composite requirements, the developers of the product were also faced with the challenge of engineering a tooth-coloured material that could be placed in 4-mm bulk increments. The incorporation of the Ivocerin light initiator has enabled the development of a composite resin that has an enamel-like appearance and the ability to be cured in bulk increments of 4-mm.

Optimization of the monomer blend: In addition to the new Ivocerin initiator described in detail in the previous chapters, Tetric EvoCeram Bulk Fill also contains camphorquinone and 2,4,6 trimethyl benzoyl diphenyl phosphine oxide (acyl phosphine oxide – comparable to Lucirin® TPO). These components are used in well-balanced proportions to ensure optimized reactivity, working time, depth of cure and strength.

Light sensitivity (working time in ambient light): Due to the incorporation of the light initiators, camphorquinone, acyl phosphine oxide and Ivocerin, Tetric EvoCeram Bulk Fill can be applied and cured in increments of 4 mm. However, it is of utmost importance to prevent premature polymerization as a result of the heightened conversion rate of the monomer. That is, an adequate working time, for placing and sculpting the restoration should be ensured. As light-cured composites generally contain photoinitiators that react to blue light, the blue light contained in ambient light or operatory light can also trigger premature polymerization in these materials (Fig. 1).

Tetric EvoCeram Bulk Fill contains a patented light sensitivity inhibitor, which prevents premature polymerization in these materials (Fig. 1). Just 1/10 of this amount is necessary in the case of the light sensitivity inhibitor. This is advantageous, as the small amount of stabilizer/inhibitor delays the polymerization process at low-level blue light, without impairing the depth of cure or any of the other polymerization properties.

Optimization of the filler composition: The filler technology incorporated in Tetric EvoCeram Bulk Fill is based on that of the clinically proven Tetric EvoCeram. In order to fulfil the desired composite resin requirements, Tetric EvoCeram Bulk Fill contains a number of different fillers (Figs 2 and 3). The fine primary

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**Fig. 1:** Sensitivity to ambient light working time of various bulk-fill composites established according to ISO 4049 (R&D Ivoclar Vivadent AG, June 2011)
particles of the fillers are responsible for the composite’s wear resistance and its excellent polishing properties, which are manifested in a smooth surface texture and high lustre. The largest particles of the inorganic fillers of Tetric EvoCeram Bulk Fill measure 3 µm. The composite fillers have a maximum size of 50 µm. In the polymerized state, they behave like the smaller inorganic primary particles. The large filler particles do not protrude from the surface. Consequently, the filling can be polished to a high-gloss finish.

Other manufacturers of bulk-fill composites mainly use coarse fillers (Figs 4 to 6). This increases the filler content and reduces the polymerization shrinkage. However, when these materials are also used as the top-most layer, they have a distinct effect on the filling’s surface texture, polishing properties, its wear behaviour and plaque resistance.

Polishing properties: The polishing properties are determined by the composition of the different-sized fillers. Tetric EvoCeram Bulk Fill contains the same type of fillers as Tetric EvoCeram. As a result, its polishing properties are just as good as those of the conventional filling material. The two composites (SonicFill from Kerr and SDR Flow from Dentsply) that contain the coarse inorganic fillers show very little shine, even after 30 s of polishing (Fig. 7).

Wear behaviour: Apart from affecting the polishing properties, the type of filler used also has a considerable influence on the wear behaviour of a composite resin. The materials containing the visibly coarser fillers are less resistant to wear than the composites featuring smaller filler particles (Fig. 8).

Shrinkage and shrinkage stress and their influencing factors: The composite filler is responsible for reducing polymerization shrinkage and lowering shrinkage stress. Tetric...
EvoCeram Bulk Fill contains a special composite filler that relieves shrinkage stress. The volumetric shrinkage of Tetric EvoCeram Bulk Fill and SonicFill after 1 h is below 2% and therefore comparable to that of conventional low-shrinkage composites (Fig. 9). The shrinkage of flowable bulk-filling composites is significantly higher at 3.3%. The composite filler acts as a shrinkage stress reliever. It is a “gentle giant”, which is capable of absorbing the shrinkage stress due to its low modulus of elasticity of 10,000 MPa compared with 70,000 MPa in conventional glass fillers. This is a particularly important factor in bulk-fill materials.

The shrinkage stress of the composite rises, as the thickness of the increments increases from 0.8 mm to 2.0 mm. Nevertheless, the in-

**Fig. 7:** Average surface gloss of five different composite filling materials compared with Tetric EvoCeram Bulk Fill after polishing with optraPol next Generation – in relation to the polishing time (R&D Ivoclar Vivadent AG, 2011)

**Fig. 8:** Average vertical wear of Tetric EvoCeram Bulk Fill in comparison with Tetric EvoCeram and other bulk-fill materials (R&D Ivoclar Vivadent AG, 2011)

**Fig 9:** Volumetric shrinkage of bulk-fill composites (*) measured with a mercury dilatometer after 60 min, or (°) according to the Archimedes principle (K. Vogel, Rheinberger V. Shrinkage and contraction force of bulk filling and microhybrid composites. AADR Abstract, 838, Florida 2012)
crease within the first few seconds of curing is lower in the thicker layers (Fig. 1). This slow build-up reduces the risk of marginal gap formation. The shrinkage stress in 4-mm increments is lower than in 2-mm layers, and the slope of the curve is much shallower than that of increments of 0.8 and 2.0 mm.

Shrinkage stress in comparison: The shrinkage stress rises in relation to the increasing thickness of the increment from 0.8 to 2.0 mm. At a layer thickness of 4 mm, the shrinkage stress ceases to increase. Furthermore, 4-mm increments of bulk-fill composites do not show higher shrinkage stress levels than conventional 2-mm layers. *Tetric EvoCeram Bulk Fill* shows the lowest shrinkage stress of all the sculptable bulk-filling materials in increments of 2 and 4 mm (Table 1). The new *Ivocerin* initiator has enabled the development of an esthetic bulk-fill material that can be applied in 4-mm increments and cured within 10 seconds. What’s more, the material is comparable to conventional direct filling composites in terms of its surface quality, working time and shade blend with natural teeth.

**Dipl.-Ing. Karin Vogel, Schaan (Liechtenstein)**

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**Table 1: Shrinkage stress of bulk-fill and conventional composites in different increment thicknesses (R&D Ivoclar Vivadent AG, February 2013)**

<table>
<thead>
<tr>
<th>Increment thickness</th>
<th>Sculptable bulk-fill composites</th>
<th>Flowable bulk-fill composites</th>
<th>Conventional composites</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Tetric EvoCeram Bulk Fill</td>
<td>SonicFill / Kerr</td>
<td>Filtek Supreme XTE / 3M Espe</td>
</tr>
<tr>
<td>0.8 mm</td>
<td>1.21 MPa</td>
<td>1.38 MPa</td>
<td>1.51 MPa</td>
</tr>
<tr>
<td>2.0 mm</td>
<td>1.51 MPa</td>
<td>1.77 MPa</td>
<td>1.93 MPa</td>
</tr>
<tr>
<td>4.0 mm</td>
<td>1.40 MPa</td>
<td>1.77 MPa</td>
<td>1.93 MPa</td>
</tr>
</tbody>
</table>

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**Fig. 10: Shrinkage force of Tetric EvoCeram Bulk Fill in different layer thicknesses (R&D Ivoclar Vivadent AG, 2011)**
Before the introduction of bulk fill composites, standard dental teaching recommended a maximum layer thickness of 2 mm for composite fillings [1, 2]. This was in order to minimise shrinkage stress and to ensure adequate depth of cure. Assuming correct, adequate curing with a suitably functioning curing unit, translucency and shade have the most significant effect on the curing depth. The darker and more opaque a composite, the lower the depth of cure [3]. The characteristics of Tetric EvoCeram Bulk Fill allow an enamel-like translucency of 15%, which ensures adequate polymerisation at depth. The inclusion of Ivocerin means that Tetric EvoCeram Bulk Fill can be applied in increments of up to 4 mm, without compromising cure or esthetics. The following internal tests conducted by Ivoclar Vivadent’s Research and Development (R&D) department, as well as external investigations confirm the effectiveness of the photoinitiator formulation with Ivocerin.

Vickers hardness

Vicker hardness testing was carried out by R&D Ivoclar Vivadent AG: Samples of each of the three Tetric EvoCeram Bulk Fill shades were cut and the Vickers hardness measured at the top and at a depth of 4 mm after removal of the inhibition layer. The values measured at the top were set to 100% and the values measured at 4 mm are expressed as a percentage of this value. Professor David Watts of the University of Manchester, UK, defined an acceptable curing depth as when the bottom hardness corresponds to at least 80% of the surface hardness [4]. Various light intensities were employed and the curing times were adjusted accordingly to ensure a similar light output in each case. For each of the shades, the 4 mm hardness value exceeded 80% of the surface hardness under all curing settings (Figs 1 to 3).

Depth of cure in comparison to other composites

In July 2012, Dr A. Rzanny and M. Fachet of Universitätsklinikum Jena, Germany, investigated the depth of cure achieved with Bluephase and Bluephase Style in Tetric EvoCeram Bulk Fill and compared it to that of other composites: Rzanny et al calculated the depth of cure of the composites: Tetric EvoCeram Bulk Fill (IVA), Venus Bulk Fill (Universal, Heraeus Kulzer) and Tetric EvoCeram (A3) by using a Penetrometer after curing for 10 seconds with Bluephase G2 (1200 mW/cm²) or Bluephase Style (1100 mW/cm²) and established their Vickers hardness values. Depth of cure measurement according to ISO 4049 yielded the following results: There was no significant difference between curing lamps for any of the composites. Both bulk fill composites Tetric EvoCeram Bulk Fill and Venus Bulk Fill far exceeded the manufacturer indicated allowable increment thickness (4 mm) in terms of depth of cure (approx. 5 mm). Tetric EvoCeram is not a bulk fill composite and is intended to be applied in 2 mm increments (Fig 4).

Evaluation of the depth of cure and surface microhardness

C. Sabatini of the Dental Biomaterials Research Laboratory, State University of New York, Buffalo, USA, evaluated the depth of cure and surface microhardness of a new bulk fill composite system in October 2012: By means of Knoop hardness tests, Sabatini investigated the depth of cure and surface mi-
crohardness of Tetric EvoCeram Bulk Fill, two further bulk fill products: x-tra fil (Voco) and SonicFill (Kerr), and Tetric EvoCeram as a control. Two light curing units were employed: Bluephase G2 (1200 mW/cm²) and Bluephase 20i Turbo (2000 mW/cm²) with exposure times of 10 and 5 seconds respectively.

Results: Two way analysis of variance (ANOVA) revealed no difference in the average bottom/top hardness values for the type of polymerisation unit used. However significant differences were found between certain restorative composites cured with the same light source (p < 0.001). When polymerised with Bluephase G2 there were no significant differences between x-tra fil, Tetric EvoCeram Bulk Fill and Tetric EvoCeram but all were significantly different to SonicFill. When polymerised with Bluephase 20i, there were also no significant differences between x-tra fil, Tetric EvoCeram Bulk Fill and Tetric EvoCeram but not significantly lower than x-tra fil. Notably both Tetric EvoCeram Bulk Fill at 4 mm and Tetric EvoCeram at 2 mm fulfilled the Watts criterion with all figures exceeding 80%. In this investigation, x-tra fil and SonicFill did not achieve 80%.

Figure 5 shows the bottom/top hardness ratios at different depths for Tetric EvoCeram Bulk Fill when cured with both Bluephase lights.

Table: Average bottom/top hardness ratios at the recommended increment thickness per material

<table>
<thead>
<tr>
<th></th>
<th>x-tra fil / Voco (4 mm)</th>
<th>SonicFill / Kerr (5 mm)</th>
<th>Tetric EvoCeram Bulk Fill (4 mm)</th>
<th>Tetric EvoCeram (2 mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bluephase G2</td>
<td>70.6 %</td>
<td>47.1 %</td>
<td>85.7 %</td>
<td>85.1 %</td>
</tr>
<tr>
<td>Bluephase 20i</td>
<td>69.4 %</td>
<td>55.6 %</td>
<td>86.9 %</td>
<td>81.4 %</td>
</tr>
</tbody>
</table>

This investigation showed that Tetric EvoCeram Bulk Fill achieved in excess of the necessary 80% bottom/top hardness ratio at a depth of 4 mm, independent of the light source. Tetric EvoCeram also achieved this at a depth of 2 mm.
The effect of curing duration on the depth of cure and modulus of elasticity

In vitro testing was carried out to evaluate the effect of curing duration on the depth of cure and modulus of elasticity in bulk fill composites [5]. S. Zawawi et al. from the Department of Restorative Sciences and Biomaterials of Boston University, Boston (MA), USA, used Vickers hardness measurements to evaluate the effect of curing duration on the depth of cure and modulus of elasticity in the bulk fill composites: Tetric EvoCeram Bulk Fill, SDR (Dentsply) and Venus Bulk Fill (Heraeus Kulzer). The Bluephase 16i (1600 mW/cm²) curing light was used to cure the composites for either 10 or 40 seconds. The Vickers hardness values for the composites at the surface and at a depth of 2 mm, 4 mm and 6 mm were determined.

In this study, Tetric EvoCeram Bulk Fill exhibited higher microhardness than SDR and Venus Bulk Fill at all depths and curing times (Fig. 6).

The mean modulus of elasticity was also measured for each bulk fill composite with both 10 seconds and 40 seconds of curing (Fig. 7). There was no significant difference between Tetric EvoCeram Bulk Fill samples when cured for 10 seconds or 40 seconds. However, there were significant differences in modulus between the different materials at both 10 and 40 seconds. Whereas curing duration had no discernible effect on the modulus of elasticity for Tetric EvoCeram Bulk Fill, this was not the case for SDR or Venus Bulk Fill — where there was a clear difference (increase) between 10 and 40 seconds polymerisation time.

Conclusion: Tetric EvoCeram Bulk Fill achieves higher mechanical properties than the other products and is almost indifferent to the length of cure (10s vs. 40s). Notably the bottom/top ratio of the Vickers hardness for Tetric EvoCeram Bulk Fill at 4 mm (10 seconds = 95.5%, 40 seconds = 98.5%) and even at 6 mm (10 seconds = 85.7%, 40 seconds = 96.2%) far exceeds the 80 % level stipulated by Watts. The studies involving Vickers hardness and Knoop hardness measurements confirmed the efficacy of the Ivocerin formulation, which means that Tetric EvoCeram Bulk Fill offers an adequate depth of cure of at least 4 millimetres.

Joanna-Claire Todd (BA, MPH), Schaan (Liechtenstein)

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LITERATURE

For decades, students of dentistry were taught that direct restorations made of composite resin should be applied into the cavity in increments. The main reason was the low depth of cure of conventional composites (one to two millimetres [mm]). Additional reasons included the belief that the incremental technique could partially offset the shrinkage, or shrinkage stress, which takes place as the composite resin polymerizes. It was said that polymerization shrinkage may result in marginal gaps, debonding from the cavity floor and/or, if a strong bond to the tooth is present, may also lead to movement of the remaining tooth structure (cusp movement/deflection), with the latter being believed to entail the risk of enamel/dentin crack formation, infractions and cusp fractures.

Imagine a composite material that fully cures in thick increments of up to 4 mm when exposed to the light of a standard curing device and in addition is very tolerant of operator errors, e.g. oblique positioning of the light probe or polymerization from a substantial distance to the filling? Such a material is indeed available and on the following pages we will show which errors this material is able to tolerate and how it behaves in the cavities of extracted teeth.

Tetric EvoCeram Bulk Fill and marginal gaps

The aim was to confirm or refute the assumption that thick composite layers result in poorer marginal quality than composites applied using the conventional incremental technique. This question was investigated with an experimental setup that used an extracted mandibular molar in which proximal occlusal cavities were prepared on both the mesial and distal side. The cavity was designed with a proximal depth of 4 mm, an occlusal depth of 3 mm and a lingual-buccal width of 5 mm (Figs 1 to 3). On one side of the tooth, Tetric EvoCeram Bulk Fill was applied in a single increment; on the other side, Tetric EvoCeram was applied in three increments: a horizontal gingival, an oblique buccal and an oblique lingual increment (Fig. 2). Each layer was light-cured for 10 seconds with a Bluephase G2 curing light (1,200 milliwatts per square centimetre - mW/cm²). Two adhesive systems were employed: the single-component etch & rinse system ExciTE F and the self-etching two-component system AdheSE.

Eight fillings were placed for each test group and all restoration margins were confined to the enamel. After the fillings had been placed, excess removed and the restoration surfaces polished, the teeth were first immersed in water for 24 hours and subsequently subjected to thermocycling (10,000 cycles between 5 degrees Celsius and 55 degrees Celsius) for 10 days. After that, replicas were produced and the marginal quality was evaluated using scanning electron microscopy (SEM) (Figs 4 and 5).

Figure 6 shows the percentage of regular margin in the axio-proximal part. It is immediately evident that a) there is no difference between the bulk and incremental technique and b) that the adhesive system, rather than the incremental technique, is the determining factor for marginal quality: The etch & rinse system, which included enamel acid...
etching with 36 per cent phosphoric acid, resulted in significantly better margins (fewer gaps and irregularities) than the self-etching system.

Figure 7 shows the cross section of a dental filling that was X-rayed using high-resolution micro-CT. The filling shows a good adaptation to the cavity margin and hardly any major air bubbles.

Given that the laboratory tests did not reveal any difference between the fillings placed with Tetric EvoCeram Bulk Fill (one increment) and Tetric EvoCeram (three increments), would it not make sense to refer to the results of clinical studies on conventionally layered composite fillings placed together with ExciTE F (or any other etch & rinse system) and AdheSE? It could be expected that Tetric EvoCeram Bulk Fill would achieve similar results.

Such studies do exist. Clinical results of up to 6 years are available for Tetric EvoCeram and ExciTE. In one study, almost 90 per cent of all the restorations were still intact after six years [1]; the main reasons for replacements were marginal caries and fractures, with marginal caries mainly occurring in patients with a high caries risk. In another study carried out by the same group of researchers, ExciTE and Tetric Ceram were used to place Class II fillings in 59 patients and this study concluded with similar results [2]: after seven years only about 10 per cent of the fillings showed marginal staining and only 2 fillings exhibited marginal caries. These results tally with the above mentioned in-vitro results. Other studies that used ExciTE and AdheSE confirmed the clinical superiority of ExciTE over AdheSE with regard to the marginal quality of posterior restorations [3-5].

If we take into account that a) clinical data on Class II restorations placed with Tetric EvoCeram and the adhesive systems ExciTE F and AdheSE are available from the Internal Clinic and that b) data from other published studies on other composite resins placed in combination with AdheSE and ExciTE confirm the reliable performance of the restorations, we may assume that the restorations placed in bulk will provide
Special feature

clinical results that are equivalent to the results achieved with Tetric EvoCeram and the incremental technique, bearing in mind, however, the limitations of laboratory tests and other physical characteristics such as flexural strength, expansion, shrinkage, etc.

How come the difference in marginal staining observed between the self-etch two-component system and the etch & rinse system was clinically smaller than the laboratory data would have led us to expect? The answer to this question is simple: In the laboratory, the margins of the fillings are evaluated far too minutely under a microscope; minor irregularities and gaps that are irrelevant for clinical performance, are discovered and recorded. If the fillings are evaluated in-vitro in the same way as the dentist does in the patient – i.e. using magnifying glasses and a sharp explorer – the difference is less pronounced (Fig. 8) [6].

Restoration margins exhibiting gaps or irregularities seldom lead to marginal caries but, in the course of time, may cause marginal staining, which may be mistaken for marginal caries and result in the filling being unnecessarily replaced. A meta-analysis of 59 clinical studies on posterior composite fillings revealed that, on average, marginal staining was diagnosed seven times more frequently than marginal caries after ten years (21 per cent of the fillings versus 3 per cent of the fillings) [7]. Furthermore, the occurrence of marginal caries was unrelated to the type of adhesive system used. Marginal caries is mostly found in patients with a high caries activity.

Conclusion

Tetric EvoCeram Bulk Fill, incorporating the innovative photoinitiator system Ivocerin, is ideally suited for the direct restorative technique because it allows cavities to be filled in large increments of up to 4 mm. Compared with the three-increment technique, 4-mm cavities filled with a single increment of Tetric EvoCeram Bulk Fill did not lead to more marginal gaps. Several studies have shown that the adhesive system, rather than the composite, is the determining factor for marginal quality. Tetric EvoCeram Bulk Fill allows for effective posterior restorations without compromises in quality.

Dr. Siegward Heintze, Schaan (Liechtenstein)

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LITERATURE


Lecturers and opinion leaders often refer to practitioner-errors with the suggestion that such mistakes lead to the insufficient polymerization of composite materials. They demonstrate that a shadow is created if the light guide is held at an angle to the composite material to cure a proximal filling (Fig. 1). Unfavorable effects are also to be expected if the distance between the light probe and filling is too large or if the light probe is not positioned precisely over the filling. Interestingly, these statements are not backed up by study results – and hardly any studies on the above-mentioned various deviations from the standard position of the light probe can be found. Only the increase in distance to the composite has been investigated in a few studies and it was found that the depth of cure decreased with an increase in distance. However, these results were to some extent affected by the curing devices used [1].

How does the Tetric EvoCeram Bulk Fill composite material behave in the presence of polymerization-related errors? To investigate this issue, we drew up a test plan to examine a variety of scenarios:

- **Test group 1:** Light probe in vertical position to the composite
- **Test group 2:** Light probe at a 40-degree angle to the composite
- **Test group 3:** Light probe in vertical position, at a distance of 5 mm to the composite
- **Test group 4:** Light probe at a 40-degree angle, at a distance of 5 mm to the composite
- **Test group 5:** Light probe in vertical position over the centre of the molar, so that both proximal sides are only partially illuminated
- **Test group 6:** Same as group 5 but with a distance of 5 mm
- **Test group 7:** Light probe in vertical position to the composite. The entire light emission window is covered with a layer of composite material (thickness: 0.8 mm).

The same experimental setup was used as for the tests on marginal adaptation (see Part 6 of this series, Figs 1 to 3). Prior to the application of the composite, a steel matrix was placed and secured with a wooden wedge. The cavity was lightly isolated with glycerine gel to ensure that the composite could be removed in a single piece. Tetric EvoCeram Bulk Fill was applied in a single increment and polymerized for 10 seconds with Bluephase G2 (1,200 milliwatts per square centimetre - mW/cm²) – in the manner specified for the test groups above. Four fillings were placed for each group. After 24 hours of dry storage at 37 °C (incubator), the composite specimens were invested in epoxy resin in a darkroom, reduced to the centre by grinding and then polished. Subsequently, the hardness profile of the entire proximal length was determined, by making indentations every 0.5 mm using a Vickers Hardness tester (Fig. 2).

The data were averaged and processed graphically. The results came as a surprise (Fig. 3). None of the test groups 1 to 6 showed cervical
hardness values below 80 per cent of the surface hardness. The exception was the group with the composite-covered light emission window. This group achieved merely 71 % of the coronal hardness value.

It can therefore be said that Tetric EvoCeram Bulk Fill undergoes complete curing even if the light probe is positioned incorrectly. Obviously, operators should use a curing light capable of generating the required light output. Additionally, the light performance should be checked regularly with a measuring device. A field test in general dental practices in Germany revealed that half of the curing lights tested failed to provide the expected performance [2]. It is equally important to ensure that the light emission window is not contaminated or covered with composite material. The same field study found that 37 % of the light probes were contaminated with composite or adhesive. To sum up, Tetric EvoCeram Bulk Fill, incorporating the new Ivocerin initiator system, allows 4-mm deep cavities to be filled with a single increment. The material is tolerant of errors related to the polymerization process. Even careless positioning of the light probe still results in the sufficient curing of 4 mm layers.

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Fig. 3: Relative Vickers hardness (in per cent) of the cervical portion of the Tetric EvoCeram Bulk Fill filling relative to the Vickers hardness of the coronal part – depending on the position of the light probe

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**LITERATURE**


Bulk-fill composites enable a more efficient and ergonomic working technique due to the fact that they can be placed in thicker increments (usually 4 mm). However, bulk-fill composites with a conventional initiator system generally comprise larger filler particles in order to counteract shrinkage and polymerization stress and they demonstrate a high level of translucency in order to allow increased layer thickness. Both material properties have a significant influence on the clinical characteristics such as surface quality and esthetics (Fig. 1).

Furthermore, such materials often require longer polymerization times to ensure sufficient curing (Fig. 2), or they react very sensitively to ambient light – limiting their processing time under operatory light (see Fig. 1 regarding the light sensitivity in the article by Karin Vogel, page 9 of DZW 41/13).

In comparison to the bulk-fill materials of other manufacturers, Tetric EvoCeram Bulk Fill shows a beneficial esthetic integration (Fig. 1). Materials with a higher translucency level do not demonstrate such good shade adaptation – a notable problem in situations with discoloured dentin.

Due to the addition of the innovative photoinitiator Ivocerin, Tetric EvoCeram Bulk Fill can be polymerized in ten seconds (Fig. 2) at a light intensity of ≥1,000 mW/cm². Tetric EvoCeram Bulk Fill is an exceptional product, featuring well-balanced properties, optimized for the posterior region. These properties are demonstrated by the excellent results attained in laboratory and preclinical tests, however only clinical studies can provide truly reliable information as to the final clinical performance.

**In-house clinical study**

In an in-house clinical investigation of Tetric EvoCeram Bulk Fill, 35 restorations (11 Class I and 24 Class II) were placed in combination with a one-bottle etch & rinse adhesive system. The restorations were assessed according to FDI criteria [6, 7], using a semi-quantitative clinical evaluation (SQUACE method), which

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**Fig. 1: Standardized Class II cavities in resin teeth with cavity floor characterized with IPS Empress Direct Color Grey and filled with various bulk-fill composites in different levels of transparency.**

(ICCDE Schaan, December 2012)
enables the documentation of marginal deficiencies in relation to the overall filling margin [8]. In the following paragraphs, the results of the clinical aspects – postoperative sensitivities, esthetic integration, polishability, occurrence of enamel cracks and initial marginal quality – are briefly described.

Postoperative sensitivity
In the majority of cases (33), defective fillings represented the preoperative situation. Only two cases involved primary caries. In 51 per cent of the cases (16) of the patients. Only in one case (3 per cent), was Caries Profunda treated. Deep lesions were discovered in 46 per cent (16) of the patients. Only in one case (3 per cent), was Caries Profunda treatment (application of a calcium hydroxide compound) necessary, due to proximity to the pulp.

Despite the fact that the lesions were partly very close to the pulp, no occurrence of postoperative discomfort, related to the applied adhesive or the filling material, was reported at baseline or in the meantime [9].

Esthetic integration
The optical properties have been optimized to meet the requirements of posterior restorations so that the shade system could be reduced to just three shades (IVA, IVB and IVW). The Iovacrin photoinitiator allows a slightly higher composite opacity compared to other bulk-fill materials due to its high quantum yield. Therefore, the translucency of the material could be adjusted in such a way that its optical properties, combined with the composite’s favourable refraction index, blend in optimally with the tooth structure (particularly enamel) (Fig. 1). Thus, virtually invisible restorations can be achieved with average-sized fillings and in cases where the dentin is not discoloured.

The examiner rated 77 per cent of the restorations as “perfect” [7, 10] and 23 per cent as “good” (minor shade deviations). These results document the material’s highly developed “chameleon effect”, especially given the fact that shade IVA was used in 80 per cent of the restorations.

Those cases in which the examiner identified slight shade deviations mostly involved either deep cavities or discoloured dentin. In such situations it is advisable to apply a thin layer of opaque material, e.g. Tetric EvoFlow Dentin, onto the dentin to obtain optimum esthetic integration (see also Figs 3 to 6).

Polishability
The clinical test also confirmed the material’s excellent polishing properties. In 77 per cent of the restorations placed in this clinical in-house study (n=35), the surface gloss was rated to be “clinically perfect” according to FDI criteria after polishing with the composite polishing instrument OptraPol Next Generation (Ivoclar Vivadent). After a period of twelve months, the results were still the same for 69 per cent.

Occurrence of enamel cracks
Thick increments can cause cracks in the enamel due to their higher volume shrinkage and especially when good adhesion to the enamel is established [11, 12]. Therefore, the clinical evaluation focused on this aspect in particular.

The majority of the treated teeth (71 per cent) already showed enamel cracks before the placement of the restoration. The position of these cracks was noted to enable them to be distinguished from subsequently occurring cracks. Cold-light optical fibres (light probe from Lercher) were used for crack detection. However, no additional cracks were identified at baseline. This confirms that the tensions caused by Tetric EcoCeram Bulk Fill are not higher than those of other posterior compos-

<table>
<thead>
<tr>
<th>FDI criteria /evaluation</th>
<th>Excellent</th>
<th>Good (excellent after correction)</th>
<th>Acceptable</th>
<th>Inadequate (repairable)</th>
<th>Unacceptable (new restoration necessary)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Postoperative sensitivity</td>
<td>35 (100 %)</td>
<td>0 (0 %)</td>
<td>0 (0 %)</td>
<td>0 (0 %)</td>
<td>0 (0 %)</td>
</tr>
<tr>
<td>Anatomic shape</td>
<td>34 (97 %)</td>
<td>1 (3 %)</td>
<td>0 (0 %)</td>
<td>0 (0 %)</td>
<td>0 (0 %)</td>
</tr>
<tr>
<td>Surface/gloss/pores</td>
<td>27 (77 %)</td>
<td>2 (6 %)</td>
<td>6 (17 %)</td>
<td>0 (0 %)</td>
<td>0 (0 %)</td>
</tr>
<tr>
<td>Esthetics</td>
<td>25 (71 %)</td>
<td>10 (29 %)</td>
<td>0 (0 %)</td>
<td>0 (0 %)</td>
<td>0 (0 %)</td>
</tr>
<tr>
<td>Surface discoloration</td>
<td>35 (100 %)</td>
<td>0 (0 %)</td>
<td>0 (0 %)</td>
<td>0 (0 %)</td>
<td>0 (0 %)</td>
</tr>
<tr>
<td>Patient satisfaction</td>
<td>34 (97 %)</td>
<td>1 (3 %)</td>
<td>0 (0 %)</td>
<td>0 (0 %)</td>
<td>0 (0 %)</td>
</tr>
<tr>
<td>Material fracture</td>
<td>35 (100 %)</td>
<td>0 (0 %)</td>
<td>0 (0 %)</td>
<td>0 (0 %)</td>
<td>0 (0 %)</td>
</tr>
<tr>
<td>Tooth integrity</td>
<td>34 (97 %)</td>
<td>1 (3 %)</td>
<td>0 (0 %)</td>
<td>0 (0 %)</td>
<td>0 (0 %)</td>
</tr>
<tr>
<td>Proximal contacts</td>
<td>33 (94 %)</td>
<td>1 (3 %)</td>
<td>1 (3 %)</td>
<td>0 (0 %)</td>
<td>0 (0 %)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>% of the entire restoration margin</th>
</tr>
</thead>
<tbody>
<tr>
<td>Marginal discoloration</td>
</tr>
<tr>
<td>Marginal deficits</td>
</tr>
<tr>
<td>Submargination</td>
</tr>
</tbody>
</table>

Table: Results of the 12-month assessment according to FDI criteria (n=35)
ites which are applied in 2-mm layers. The results of this clinical study correspond with the preclinical data presented earlier. As it can be assumed that the water absorption of the composite compensates for the polymerization stress in a relatively short time [13], this baseline result can be considered reliable.

**Initial marginal quality**

Tetric EvoCeram Bulk Fill restorations demonstrated excellent marginal quality at baseline as well as at the 12-month recall. With regard to marginal discoloration, submargination and marginal irregularities, 99 per cent of the examined marginal sections were rated as “clinically perfect”. A summary of the clinical results at the 12-month recall is displayed in the table above.

**Conclusions**

The preliminary clinical data of Tetric EvoCeram Bulk Fill confirm the good results of the preclinical tests. Due to the photoinitiator Ivocerin, the material can be applied in 4-mm layers, with esthetic properties that meet the requirements of posterior restorations.

The increased curing depth of Tetric EvoCeram Bulk Fill gives the user more freedom without loss of quality. Dentists can restore cavities quickly with horizontal 4-mm increments or — perhaps even more importantly — they can adapt their layering technique to individual anatomic situations, their ergonomic preferences (e.g., the centripetal build-up technique for restorations) [14] or to esthetic aspects.

**Dr med. dent. Arnd Peschke,**

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**L I T E R A T U R E**


**Figs 3 to 6: Arnd Peschke**
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