



# Posterior Restorative

Scientific Compendium  
(For individual attention only)  
2003-06-16

## Table of contents

<b>1</b>	<b>Introduction .....</b>	<b>3</b>
1.1	<b>QUIXFIL Technology .....</b>	<b>4</b>
1.1.1	Resin Chemistry.....	4
1.1.2	Filler Technology .....	4
<b>2</b>	<b>Product Description.....</b>	<b>6</b>
2.1	Indication for Use .....	6
<b>3</b>	<b>Physical Properties of QUIXFIL .....</b>	<b>7</b>
3.1	Materials Evaluated.....	7
3.2	Polymerisation Shrinkage.....	7
3.2.1	Volume Shrinkage .....	7
3.2.2	Shrinkage Stress.....	10
3.3	Expansion in Water.....	11
3.4	Depth of Cure .....	13
3.5	Wear resistance .....	14
3.5.1	The Leinfelder Wear Test.....	14
3.5.1.1	Measurement at DENTSPLY DeTrey.....	14
3.5.1.2	Wear Measurement at Creighton University (M. Latta).....	15
3.5.2	The ACTA Wear Test (DeGee).....	16
3.6	Yield Strength and Compressive Strength .....	17
3.7	Flexural Strength and Modulus .....	18
3.8	Surface hardness .....	19
3.9	Radiopacity .....	20
3.10	Adhesion .....	21
3.11	Working Time .....	22
3.12	Flexural Fatigue Limit.....	23
3.13	Microleakage in Class II Cavities .....	24
3.14	Polishability and Toothbrush Abrasion .....	25
<b>4</b>	<b>Step-by-Step Instructions .....</b>	<b>27</b>
<b>5</b>	<b>Summary of Clinical Studies .....</b>	<b>29</b>
5.1	Clinical Investigation of the restorative system k-0112 (QUIXFIL) and Xeno III for Class I and II restorations of the University of Hong Kong .....	29
5.2	Clinical Investigation of the restorative system K-0112 (QUIXFIL) and Xeno III for Class I and II restorations at the University of Munich.....	33
<b>6</b>	<b>References .....</b>	<b>35</b>

# 1 Introduction

QUIXFIL is a light cured composite restorative which has been specifically designed for use for use in posterior restorations. Its excellent handling characteristics, high depth of cure, and low shrinkage make it fast and cost effective to place. The high radiopacity of QUIXFIL means that is also easily detected radiographically.

QUIXFIL is presented in compules for easy application, and this is complemented by its use with reliable and simple adhesive systems such as Prime&Bond NT or the self-etching adhesive Xeno III.

The physical properties and low wear characteristics of QUIXFIL rival and improve upon those of the best composites, and overall QUIXFIL provides an outstanding combination of properties that are not met by any other posterior restorative.

- Fast placement
  - high depth of cure
  - low shrinkage
  - short cure time
- Excellent handling characteristics
  - non sticky
  - sufficient working time in ambient light
  - easily extrudable from a compule
- Physical properties suitable for posterior use
  - resistance to wear
  - resistance to deformation under occlusal stress.

Together, these properties make QUIXFIL the material of choice for situations where amalgam would previously have been used.

## **1.1 QUIXFIL Technology**

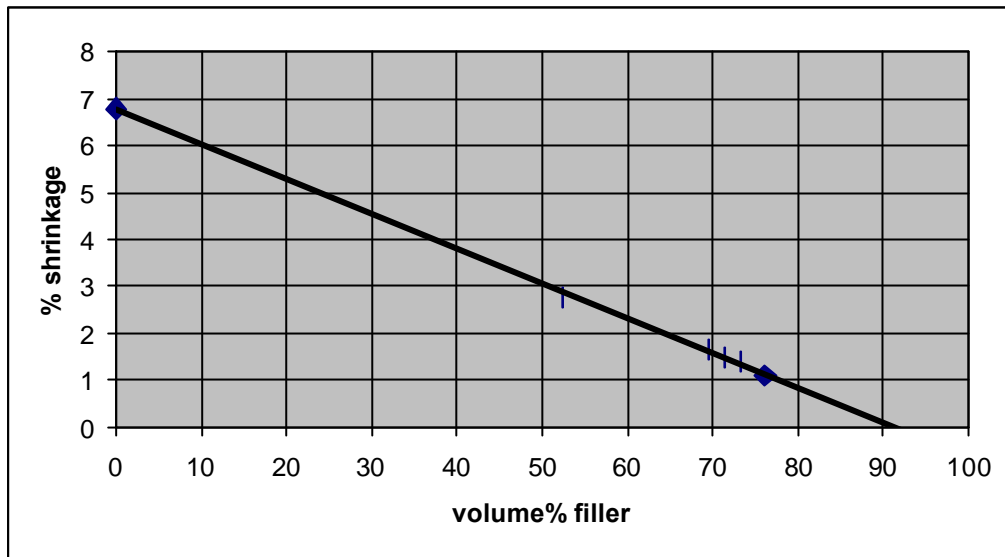
### **1.1.1 Resin Chemistry**

The QUIXFIL matrix comprise a mixture of several well known and tried methacrylate resins including ethoxylated bisphenol-A-dimethacrylate (Bis-EMA), urethane resin (UDMA), triethylene glycol dimethacrylate (TEGDMA), and trimethylolpropane trimethacrylate (TMPTMA). A small amount of butane-1,2,3,4-tetracarboxylic acid, bis-2-hydroxyethyl methacrylate (TCB) resin is also included, and this serves to give the resin mixture a high cohesion, and to reduce its hydrophobicity. These two properties help to give QUIXFIL its uniquely excellent handling properties. The matrix also contains a combination of the photoinitiator camphor quinone and the accelerator dimethylaminobenzoic acid ethyl ester, and the concentrations of these have been carefully optimised to provide a long clinical working time (reduced sensitivity to ambient light) as well as high depth of cure.

### **1.1.2 Filler Technology**

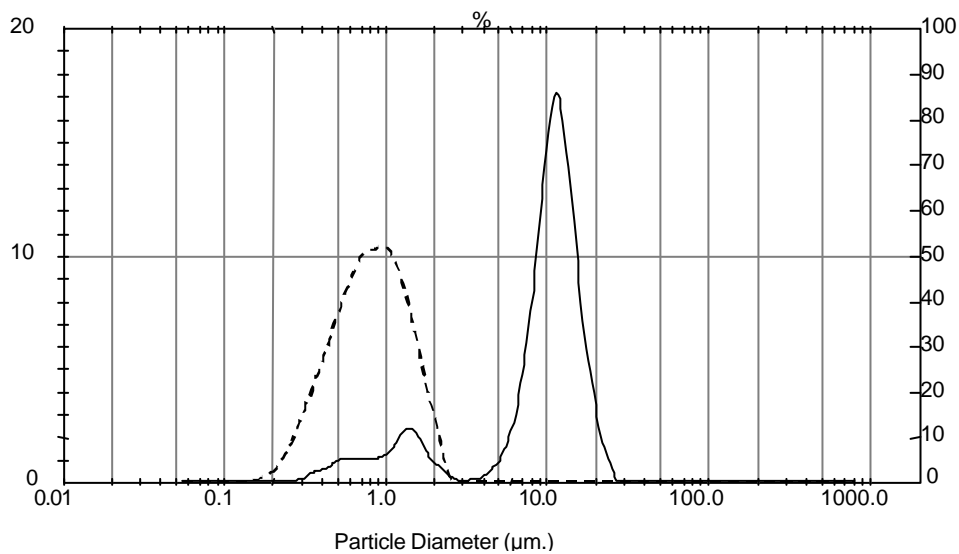
The filler component of QUIXFIL is the heart of the new posterior restorative. Consisting of two quite separate glass fractions, this patented filler allows an exceptionally high filler loading of 66% by volume (about by 86% weight) with an extrusion force from the compule under 100 Newtons. For comparison, normal composites have a filler loading around 50% by volume, with an extrusion force from the compule ranging from 100 to 200 Newtons. The new material QUIXFIL therefore has a filler content about 30% higher than most other composites, but is still easily extruded from a compule. The main consequences and advantages brought by the high filler content are an increase in the surface hardness and a reduction in the polymerisation shrinkage. The effect on shrinkage is seen in Figure 1 which shows the effect of increasing filler content (internal data). Notice that the graph does not pass through zero shrinkage at 100% filler loading as might be expected, but at just over 90% filler loading, the other approximately 10% representing the space "lost" between the filler particles.

This space has to be filled with resin before any effect is seen. Also as shown in the graph, the resin matrix itself has a shrinkage of about 6.8%.



**Figure 1** Volume % Shrinkage vs. Volume % Filler Loading for the QUIXFIL Matrix

The particle size distributions of the two glass fractions are also of interest, because these make the high filler loading possible. As shown in Figure 2, the smaller glass has a mean particle size of about 1 $\mu$ m, while the larger glass fraction has a mean particle size of about 10  $\mu$ m.



**Figure 2** Particle Size Distribution of the QUIXFIL Filler (--- 1  $\mu$ m fraction, — 10  $\mu$ m fraction)

Unlike some other composites that contain coarser glass, the 10 µm fraction has 90% under 16 µm, and no particles over 26 µm. Although the surface roughness due to the 10 µm fraction is obviously slightly higher than that of other composites which contain only small glass particles (normally less than 3 µm), it is nevertheless fully acceptable as shown in chapter 3.14 'Polishability and Toothbrush Abrasion'.

## **2 Product Description**

QUIXFIL is a resin-based restorative material specially designed for posterior use.

QUIXFIL allows a simplified and fast application technique without compromising safety and efficacy.

QUIXFIL utilises a new filler technology resulting in a unique high filler load.

QUIXFIL exhibits wear resistance and physical properties suitable for occlusal stress-bearing restorations.

QUIXFIL is available in pre-dosed Compules<sup>®</sup> tips for direct intra-oral application.

QUIXFIL is available in one universal shade.

QUIXFIL is used following application of Xeno<sup>®</sup> III Single Step Self-Etching Adhesive, Prime&Bond<sup>®</sup> NT, or other adhesives designed for use with QUIXFIL.

### **2.1 Indication for Use**

QUIXFIL is indicated for use as a direct restorative for all cavities in posterior teeth.

### 3 Physical Properties of QUIXFIL

#### 3.1 Materials Evaluated

The following composite materials were selected in our *in-vitro* competitive property evaluations. Note that during the product development and the *in-vitro* studies QUIXFIL has been designated as K-0112:

Product	Manufacturer
Filtek™ Z250	3M™
Tetric® Ceram	Vivadent
SureFil™	DENTSPLY

**Table 1** Reference Composite materials used for *in-vitro* Studies

#### 3.2 Polymerisation Shrinkage

##### 3.2.1 Volume Shrinkage

**Investigators:** U. Soltesz, Fraunhofer-Institut für Werkstoffmechanik, Freiburg  
D. Watts, Univ. Manchester  
A. DeGee, ACTA Amsterdam

**Clinical Relevance:** Excessive polymerisation shrinkage of a composite may contribute to the marginal microleakage of a restoration, and also to stress on the tooth cusps. Both of these can lead to post-operative sensitivity, and in extreme cases build-up of stress can lead to fracture of the tooth.

Polymerisation shrinkage is a consequence of the repulsive forces between individual monomer molecules, and the reduction in these repulsive forces when the monomer molecules join together to form polymers. Thus when two or more monomer molecules join together, less volume is occupied by the oligomer or polymer than by the individual molecules. Conventionally, research towards lower shrinking restorative materials has therefore concentrated on finding monomers or monomer mixtures which exhibit lower shrinkage. However, this is often accompanied by a lower degree of cross-linking, the consequence of which is lower physical properties. An alternative way of reducing shrinkage is simply to use more filler – but until the

development of QUIXFIL this was not possible without producing a paste which was too stiff to use. The patented filler combination used in QUIXFIL now makes a high filler loading AND low extrusion force with excellent handling properties possible for the first time.

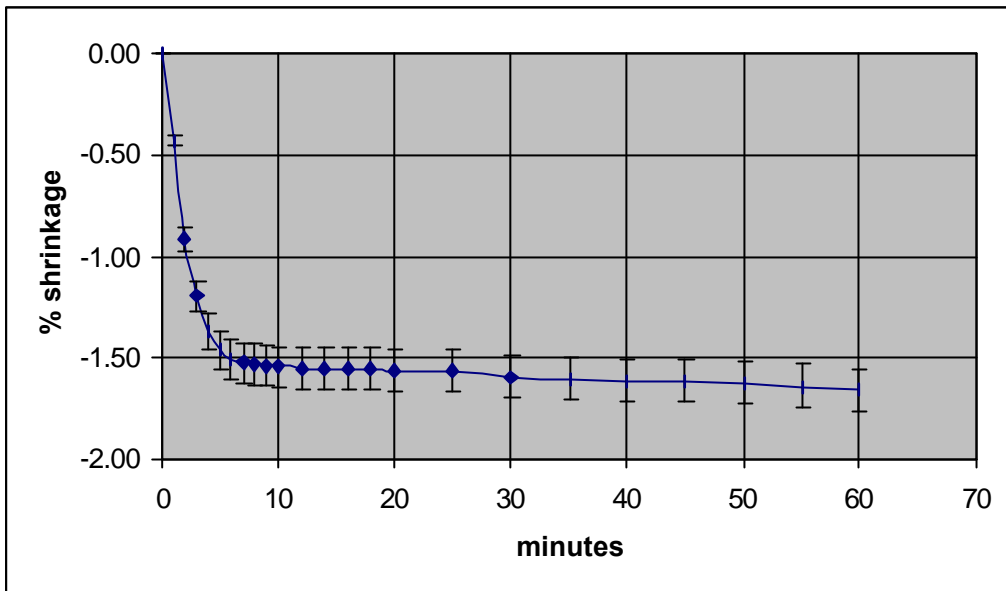
The polymerisation shrinkage of dental composite materials is easily measured and several methods are employed<sup>1-6</sup>. The shrinkage of QUIXFIL has been measured at the Fraunhofer Institute in Freiburg, at the University of Manchester, and at ACTA in Amsterdam as well as at DENTSPLY DeTrey. In both Freiburg and Konstanz a method based on the Archimedes principle was used which allows a direct measurement of volume change on cure. At DENTSPLY DeTrey water was used as the suspending medium while at the Fraunhofer Institute silicon fluid was used.

In Manchester, the volume shrinkage of QUIXFIL was measured using the Linometer method developed there. The samples were irradiated at 550 mW/cm<sup>2</sup> for 40 s, and shrinkage was measured over a period of 1 hour. After this time the polymerisation shrinkage was found to be 1.73 vol.% at 23 °C, thus confirming the results obtained by Archimedes method. Values are given in Table 2. Throughout this manual unless otherwise noted, the standard deviations are given in brackets.

<b>Material</b>	<b>Vol. % Shrinkage Fraunhofer Institute</b>	<b>Vol.% Shrinkage DENTSPLY DeTrey</b>	<b>Vol.% Shrinkage Manchester</b>	<b>Literature Values<sup>6</sup></b>
QUIXFIL (K-0112)	1.66 (0.1)	1.70 (0.05)	1.73 (0.04)	
Tetric Ceram		2.75 (0.05)	2.66 (0.2)	2.9
Filtek Z250		2.00 (0.05)		2.2
Z 100			2.54 (0.4)	

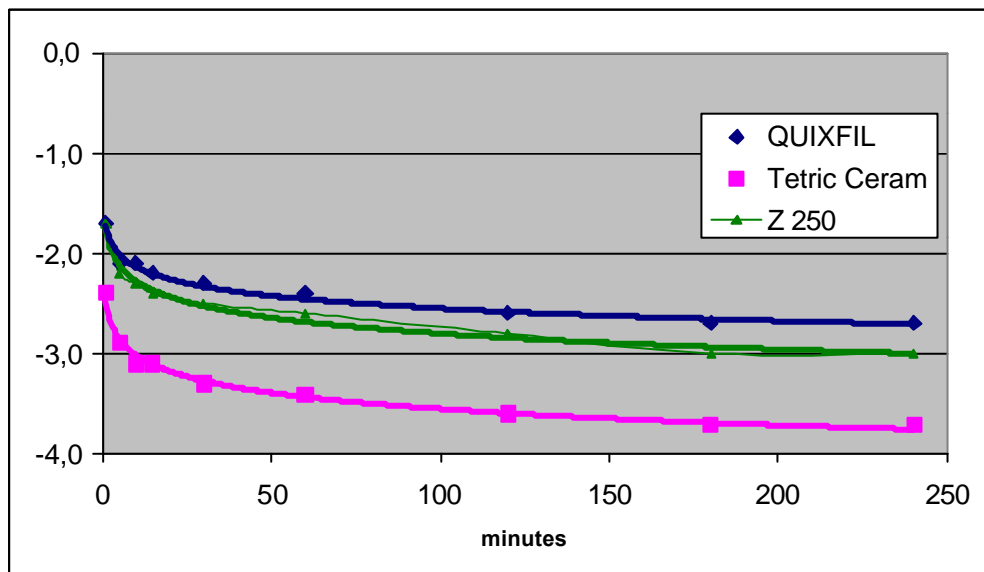
**Table 2** Volume % Shrinkage Measured by Various Methods

For the literature values quoted, a method involving a laser interferometer was used. In spite of these different methods the results are very close, giving confidence that the results are correct and reliable.



**Figure 3** Shrinkage of QUIXFIL vs. Time after Polymerisation (Fraunhofer Institute)

The shrinkage was also measured in ACTA using a dilatometer. The materials were cured for 40 seconds using a lamp with an output of 750mW/cm<sup>2</sup>, and the shrinkage was followed continuously for four hours. The results are shown graphically in Figure 4 below.



**Figure 4** Shrinkage of QUIXFIL vs. Time after Polymerisation (ACTA)

The shrinkage values found at ACTA for all three materials are higher than those found at the Fraunhofer Institute, DENTSPLY DeTrey, or Manchester, but the ranking order remains the same as determined by the Archimedes or laser method. Again, QUIXFIL was found to have the least shrinkage, followed by Z250 and then Tetric Ceram.

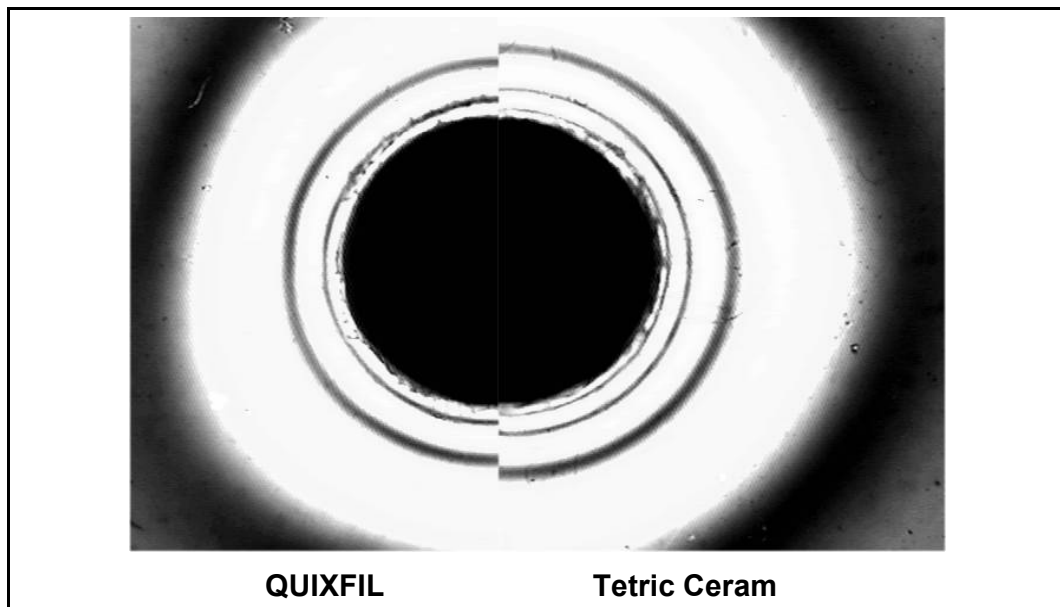
### 3.2.2 Shrinkage Stress

**Investigator:** C.-P. Ernst, Univ. Mainz

**Clinical Relevance:** A high shrinkage stress is undesirable because this can lead to post operative pain, and in extreme cases to cracking of the tooth cusps.

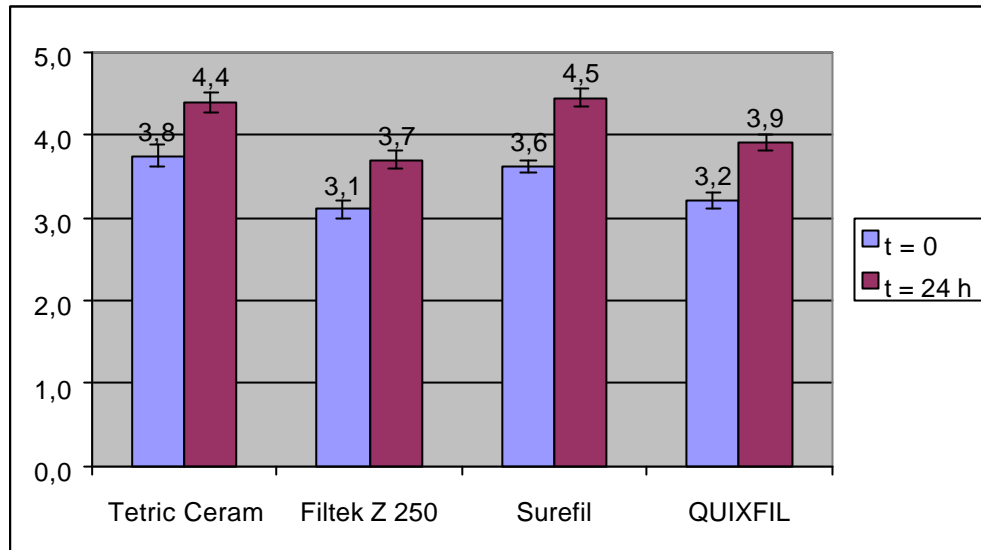
The actual stress on the walls of the tooth developed due to polymerisation shrinkage of the restoration depends not only on the degree of shrinkage, but also on other factors such as the elastic modulus of the composite material. In this method, the shrinkage stress is measured directly in a clinically relevant manner. Plates 40 mm x 40 mm x 3 mm thick and having a hole 5 mm in diameter in the centre are prepared from Araldite B resin. The inner surfaces of the holes are treated to ensure bonding of the Araldite to composite, and the holes are then filled with the composite of interest. This is cured for 60 seconds at 800 mW/cm<sup>2</sup>. Shrinkage of the composite material produces stress within the Araldite plate, and concentric isochromatic rings become visible as shown in Figure 5.

Figure 5 shows half of the resulting diagrams for QUIXFIL and Tetric Ceram superimposed in one picture. For QUIXFIL, the outer broad zero order ring is partially visible, followed by the first order ring and second order ring. For Tetric Ceram, zero, first, second and third order rings are visible. The first and second order rings for Tetric Ceram are clearly seen to be larger than those of QUIXFIL, indicating higher stress.



**Figure 5** Isochromatic Rings (dark) within the Araldite Plate indicating Polymerisation Stress

As shown in Figure 6 the shrinkage stress of QUIXFIL both immediately after curing and after 24 hours was similar to that of Z250. In contrast, both Tetric Ceram and SureFil showed significantly higher shrinkage stress at both time intervals. The low shrinkage stress obtained with QUIXFIL is further evidence of the true low shrinkage obtained with this new material.



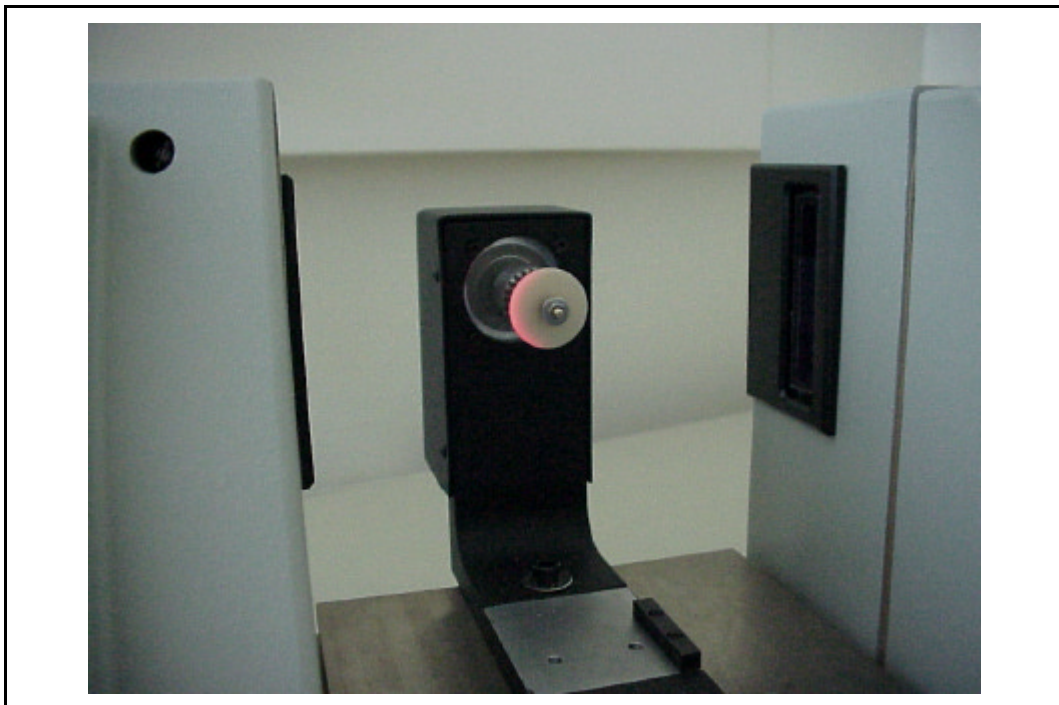
**Figure 6** Shrinkage Stress immediately and 24 h after Polymerisation (Ernst, Univ. Mainz)

### 3.3 Expansion in Water

**Clinical Relevance:** Although a small degree of expansion helps to provide stress relaxation, too great an expansion can lead to even higher stress on the tooth cusps with concomitant post-operative pain and even cusp fractures. With a material intended for use also in large posterior cavities it is therefore especially important that the expansion is not too large.

It is well known that composites shrink on curing but perhaps less well known that they also show varying degrees of expansion due to absorption of water<sup>7,8</sup>. The ISO 4049 7.12 specification refers to a “water uptake” measurement, but the direct measurement of expansion is probably a more relevant and useful test.

The expansion values below were measured in DENTSPLY DeTrey using a laser micrometer to measure the diameter of a disc in a slight modification of the method described by Martin and Jedynakiewicz<sup>7</sup>. Discs of the material 25 mm diameter and 1 mm thick are made and a small hole is bored approximately in the centre to allow the disc to be held in the micrometer. The discs are then stored dry for 24 hours to allow post cure to occur. The diameters of the discs are next measured at one hundred points around the circumference using the laser micrometer fitted with a stepping motor to rotate the disc in known increments. Finally the discs are stored in water at 37°C and the diameters of the discs are re-measured at suitable intervals until no further change in diameter takes place. The linear expansion can then be calculated and converted to volume expansion.

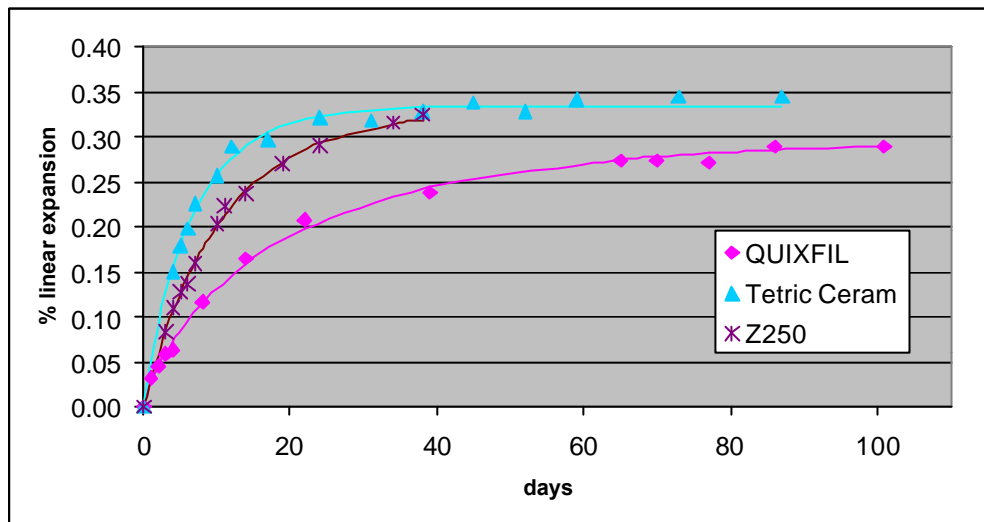


**Figure 7** An Expansion Disc being Measured with a Laser Micrometer

Commonly used dental composites have a volume expansion<sup>7</sup> of around 1%, and all the materials in the Table 3 are in this range. Due to the low resin content of QUIXFIL, its expansion is slightly lower than that of the other two composites.

Material	Volume Expansion % in Water
QUIXFIL	0.89 (0.05)
Tetric Ceram	1.00 (0.05)
Filtek Z250	0.99 (0.05)

**Table 3** Volume % Expansion after Water Absorption (saturation)



**Figure 8** Linear Expansion in Water

### 3.4 Depth of Cure

**Clinical Relevance:** Combined with a low shrinkage, a high depth of cure means that bulk placement of the material is possible.

A material with a high depth of cure but also normal or high shrinkage is of little advantage since the gap formation and polymerisation stress become too great. Only in combination with low shrinkage is a high depth of cure really useful (it was shown in section 3.2 that QUIXFIL has a volume shrinkage of only about 1.7%). The depth of cure was measured according to the method in ISO 4049. In this, material to be tested is filled into a stainless steel mould with a diameter of 4 mm, and cured with a dental lamp.

The mould is then opened, and soft material scrapped away with a plastic instrument. The length of the remaining composite post is measured, and the length is divided by two to give the ISO depth of cure. All the measurements below were made with a dental lamp having an output of 800 mW/cm<sup>2</sup>.

Material	Cure Time (s) at 800mW/cm <sup>2</sup>	Depth of cure (mm)
QUIXFIL (U)	10	4.4
Tetric Ceram (A2)	10	2.1
Filtek Z250 (A2)	10	2.6

**Table 4:** Depth of Cure according to ISO 4049

QUIXFIL therefore has a depth of cure more than twice that of Tetric Ceram, and significantly larger than that of Z250.

### 3.5 Wear resistance

**Clinical Relevance:** A low wear rate means that enamel-restorative margins and contact points remain at the correct level, and that gross loss of material does not occur. It goes without saying that a low wear rate is a prerequisite for a modern composite.

The wear rate of QUIXFIL was initially measured in DENTSPLY DeTrey using the method developed by Leinfelder, and then at ACTA using the method developed there<sup>9</sup>. As an additional check, the wear rate was measured again at Creighton University using the Leinfelder method.

#### 3.5.1 The Leinfelder Wear Test

##### 3.5.1.1 Measurement at DENTSPLY DeTrey

In a slight modification of the Leinfelder test<sup>11</sup>, the composite materials are first set in a hard silicon putty. This allows very slight movement of the sample as with a natural tooth. After ageing the sample in water at 37°C for one week it is placed under a conical steel piston in a slurry of polymer beads. The piston is driven up and down with a twisting action, so that the overall effect is a repeated initial percussion followed by a grinding action between the test material and the steel piston, with the polymer beads acting as food substitute.

The force applied by the piston is accurately regulated to between 115 N and 120 N, and 200000 cycles are normally carried out. Several methods can be used to assess the resulting wear, and the results given are the average diameter of the depression produced in the specimen.

QUIXFIL	1.09 (0.03) mm after 200000 cycles
Tetric Ceram	1.21 (0.05) mm after 200000 cycles
Filtek Z250	0.98 (0.05) mm after 200000 cycles

According to the Leinfelder test, the wear rate of QUIXFIL is therefore essentially the same as Filtek Z250, and both have significantly lower wear than Tetric Ceram.

### 3.5.1.2 Wear Measurement at Creighton University (M. Latta)

The Leinfelder test was also used at Creighton, albeit under slightly different conditions to those used in Konstanz. It is quite normal that different test centres use slightly different conditions, and contrary to popular conception this should be seen positively in that overall the materials are tested under a wider spectrum of conditions. In Creighton for example, the test specimens were aged for 24 hours rather than 7 days. Further differences are that the pistons were set to load the test specimens with 80 Newtons, and the wear was evaluated after 400,000 load cycles by measuring volume loss and the maximum depth of the cavity. Twelve samples each of QUIXFIL and Tetric Ceram were tested, and the results are shown in Table 5:

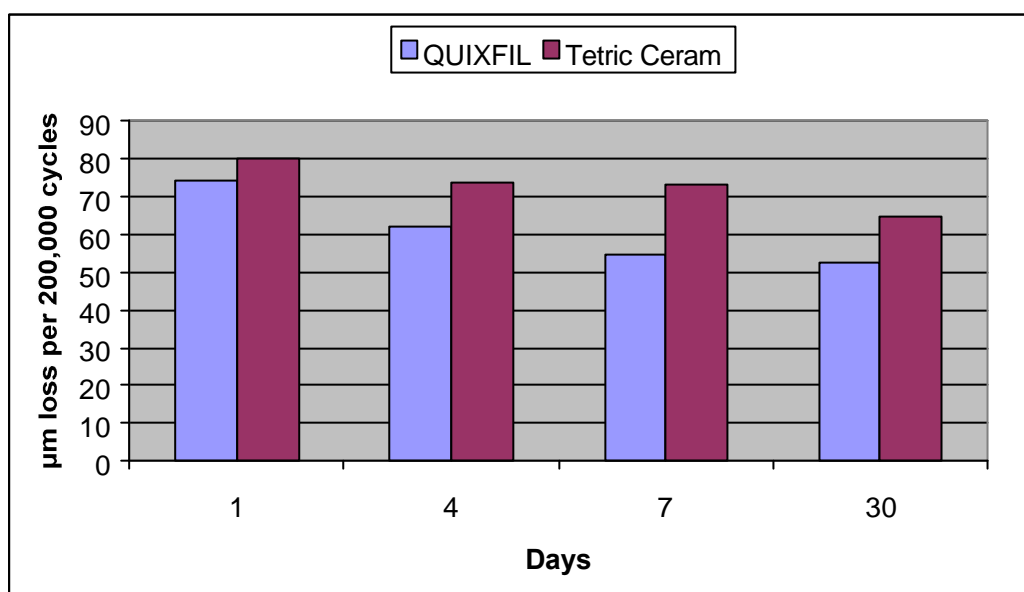
Material	Volume Loss (mm <sup>3</sup> )	Maximum Depth of Cavity (µm)
QUIXFIL	0.027 ± 0.008	103.2 ± 17.3
Tetric Ceram	0.064 ± 0.006	153.5 ± 17.3

**Table 5** Leinfelder Wear Results (M. Latta, Univ. Creighton)

From the table above it is clearly seen that the mean volume loss of QUIXFIL is less than half that for Tetric Ceram – 42% in fact. This lower wear rate of QUIXFIL is similarly shown by the maximum depth of the cavities worn, and confirms the results found by DENTSPLY DeTrey.

### 3.5.2 The ACTA Wear Test (DeGee)

With the ACTA test, materials are set in a wheel which is rotated against an antagonist wheel at a speed of one revolution per second in the presence of a slurry of ground rice and poppy seeds. The pressure between the two wheels is adjusted to 15 Newtons, and the slip rate between the wheel containing the test material and the antagonist wheel is set to 15%. In this way, the organic material is drawn between the two wheels and acts as an abrasive. The material loss is measured with a profilometer at intervals of 200,000 cycles, and at time intervals of 1 day to 2 months after polymerisation.



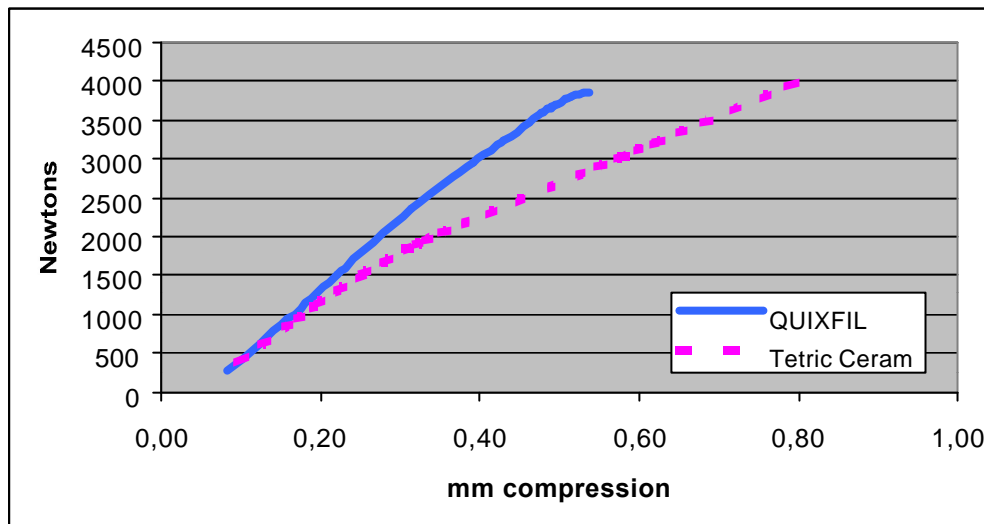
**Figure 9** Rate of QUIXFIL and Tetric Ceram measured at ACTA 1 to 30 days after Polymerisation

At all time intervals, the wear rate of QUIXFIL is shown to be consistently lower than that of Tetric Ceram, confirming the results found at DENTSPLY DeTrey and in Creighton using the Leinfelder wear test.

### 3.6 Yield Strength and Compressive Strength

**Clinical Relevance:** The yield strength of a dental composite is especially important, because this indicates the force that the material can withstand BEFORE damage occurs.

The yield strength of a material is defined as the load at which the stress-strain relationship of the material becomes non linear. Because the non linear behaviour is due to plastic flow and crack formation within the material, the yield strength represents the highest load to which a material can be subjected before a permanent change in shape and structural damage occurs. A typical yield point determination curve for QUIXFIL and Tetric Ceram is shown below.



**Figure 10** Yield Strength Determination of QUIXFIL and Tetric Ceram

From Figure 10 it can be seen that the compression curve for QUIXFIL is linear up to about 2500 Newtons, while the curve for Tetric Ceram deviates from the initial gradient at around 1500 Newtons, corresponding to yield strengths given in Table 6.

The yield point is a very important property for dental materials, since neither flow nor crack formation are desirable, and it is important to know the load at which these start, rather than when they end as measured by the compressive strength. It is therefore clear that the yield strength of a material should be higher than the loads applied during use, and in a material intended for use in posterior teeth this is especially important. Due to the unique particle size distribution of the fillers used in QUIXFIL, the yield strength of QUIXFIL is 66% and 26% higher than that of Tetric Ceram and Filtek Z250 respectively. Although the catastrophic failure point (compressive strength) of Tetric Ceram and Filtek Z250 are both higher than that of QUIXFIL, both are therefore permanently damaged at far lower loads than QUIXFIL.

<b>Material</b>	<b>Yield strength (MPa)</b>	<b>Compressive strength (MPa)</b>
<b>QUIXFIL</b>	<b>191 (8.0)</b>	278 (10.0)
<b>Tetric Ceram</b>	<b>115 (7.0)</b>	360 (15.0)
<b>Filtek Z250</b>	<b>151 (7.0)</b>	380 (45.9)

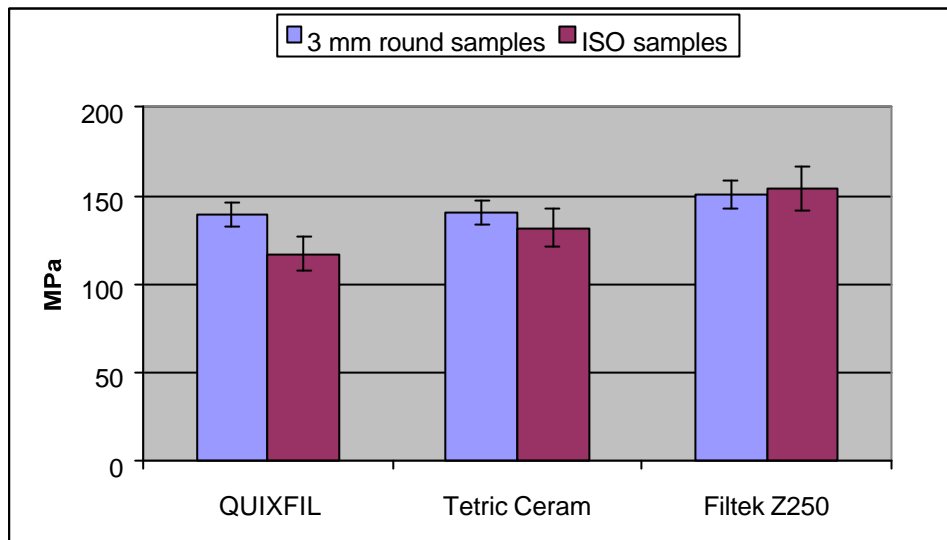
**Table 6** Yield Strength and Compressive Strength (internal results)

### 3.7 Flexural Strength and Modulus

**Clinical Relevance:** The flexural strength of a dental material is an important property since materials may be used in thin layers or in poorly supported edges where flexural forces occur. A high flexural strength is therefore needed. It is also important that the modulus is in a suitable range, since together with the yield strength this determines the degree of brittleness of a material. A material in which the modulus is too high will be brittle, while if the modulus is too low, deformation and flow can occur.

The flexural strength was measured according to ISO using samples nominally 2 mm square and 25 mm long. However due to the need to remove excess material by sanding, scratches and malformations are introduced which can lead to false values and high variations.

Therefore the flexural strength was also measured according to a literature technique in which the samples are formed in 3 mm diameter glass tubes. In this case cylindrical samples free of any defects are produced, and the values found by this method are therefore slightly higher with lower variation than those found by the ISO method. However as shown in Figure 11, there is no statistically significant difference between the flexural strengths for the three materials measured using either method. Likewise, the flexural moduli for all three materials have been found to be  $10000 \pm 1000$  MPa, and all are in the acceptable range.



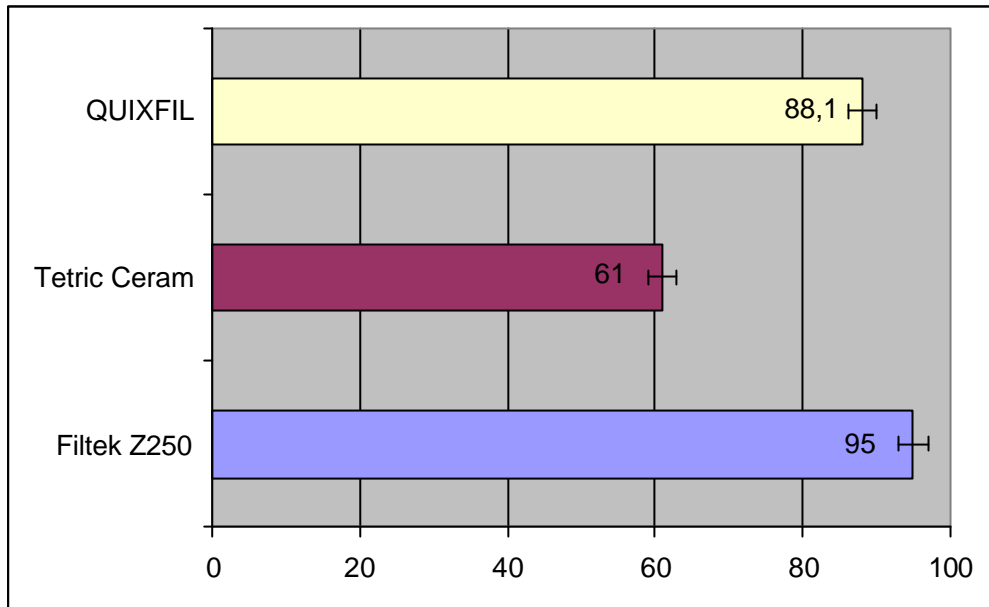
**Figure 11** Flexural Strength

### 3.8 Surface hardness

**Clinical Relevance:** The surface hardness of a restorative material is a measure of its resistance to surface indentation, and therefore has some correlation with wear resistance.

Several methods are used for measuring the surface hardness of a material, and each has advantages in some circumstances. Perhaps the simplest method is known as the Barcol hardness, which involves pushing a needle under spring loading into the material to be tested. The hardness is then proportional to the depth of penetration of the needle and can be read directly from a dial. Although this method is very quick, the readings can be variable for composite materials if the size of the point is similar to or smaller than that of the filler particles. This problem is largely overcome by the Vickers hardness method in which a diamond pyramid is pushed into the surface of the test material under a known load. The size of the resulting depression is measured and is converted to hardness values with the use of tables.

The Vickers hardness method was therefore used in DENTSPLY Konstanz. The hardness values given below were measured with a load of five kilograms (49.03 Newtons) and by convention are referred to as the “HV5 value”.



**Figure 12** Vickers Hardness (HV5)

The surface hardness of QUIXFIL and Z250 are therefore very similar, but that of Tetric Ceram is significantly lower.

### 3.9 Radiopacity

**Clinical Relevance:** The radiopacity of a restorative has to exceed that of the enamel and dentine in order to be visible with standard X-ray procedures. In general, the higher the radiopacity of a restorative, the more easily discernible it is.

The radiopacity of QUIXFIL and the competitive materials was measured relative to aluminium according to ISO 4049 section 7.14. The transmission of each region of the exposed and developed film was measured at 500 nm using a visible spectrometer, and the radiopacity of each material was calculated from the resulting calibration line.

Dentin has a radiopacity equivalent of about 1 mm of Al, while enamel is equivalent to about 2 mm of Al<sup>10</sup>. Due to its high filler content, QUIXFIL has a radiopacity equivalent to 4.0 mm of Al.

### 3.10 Adhesion

**Clinical Relevance:** Strong adhesion to tooth substrate is needed to prevent microleakage, and in today's climate of conservative dentistry, to hold the restorative in place in the absence of mechanical interlocking.

Adhesion samples were prepared using QUIXFIL and Xeno Bond III with unetched tooth substance, or QUIXFIL and Prime & Bond NT with etched tooth substance. In each case the instructions in the respective DFUs were followed. After preparation, the samples were stored overnight in water at 37°C before being thermocycled 1800 times between 5 and 55 °C.

	<b>Xeno III</b>	<b>Prime&amp;Bond NT</b>
Dentin	18.8 (1.1) MPa	22.0 (3.8) MPa
Enamel	21.8 (1.7) MPA	30 (4.3) MPa

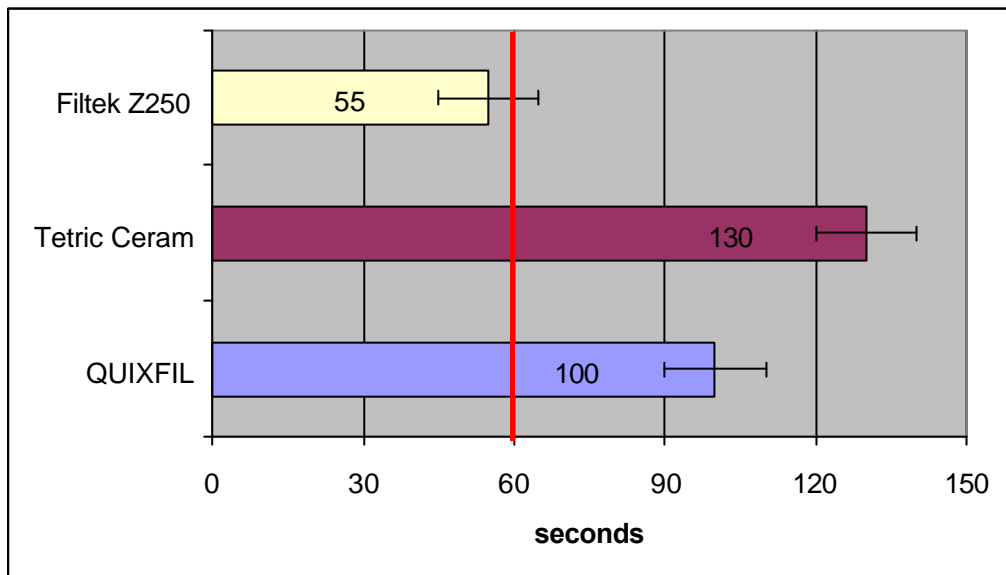
**Table 7** Adhesion of QUIXFIL

The adhesion to both dentin and enamel is satisfactory using both adhesive systems. The adhesion to dentine is essentially the same for both systems, while with enamel, a separate etching step inevitably brings some advantage at the cost of a longer treatment time.

### 3.11 Working Time

**Clinical Relevance:** Adequate working time of a composite is needed so that clinicians have time to complete the necessary procedures.

The lifetime of a light cured dental filling material refers to the time that the material is likely to remain workable under the lighting conditions in a dental surgery. A standard brightness of 10000 lux was initially chosen in the method developed for ISO 4049, though this has since been reduced to 8000 lux. However materials in this report were tested under the harsher conditions of 10000 lux in order to have a higher margin of safety.



**Figure 13** Working Time at 10000 lux

While a working time sufficiently long to allow the dentist time to place and form the filling is needed, an excessively long working time serves no useful purpose. The ISO 4049 specifies 60 seconds at 8000 lux as the minimum permissible working time, although a slightly longer working time is desirable to ensure that sufficient time is also available under stronger lighting conditions. Both QUIXFIL and Tetric Ceram therefore have a sufficiently long working time, while that of Filtek Z250 is rather short.

### 3.12 Flexural Fatigue Limit

**Investigator:** Prof. Marc Braem, University of Antwerp, Belgium

**Clinical Relevance:** Many tests, such as compressive or flexural strength, involve simply increasing a load on a test specimen until failure occurs. However the high forces often reached in the laboratory rarely occur clinically, and it is more relevant to know how the material behaves under repeated loads that are less than those needed to produce instant catastrophic failure. The fatigue limit is such a test, and is a measure of a materials resistance to fracture through repeated stress at levels that do not lead to immediate fracture.

**Method:** The fatigue resistance of a material can essentially be determined in two modes. In the first mode, samples of the material are repeatedly subjected to a fixed load until failure of the specimen occurs. In order to obtain statistically significant results however a large number of specimens is required, and depending on the force chosen and the fatigue resistance of the material it is possible that a large number of cycles will also be needed. In the second method, the number of load cycles for each experimental series is fixed, and the load is increased in successive experiments until 50% of the specimens under test fail within the chosen number of cycles. This second method was used in the present test, and the specimens were subjected to 10,000 cycles at various loads.

**Test Details:** The fatigue specimens comprised beams of material 1.2 mm deep, 5 mm wide and 40 mm long. These were kept in water at 37°C for 30 – 2 days before being tested and were also kept wet at 37°C during the test. For testing, the specimens were clamped between parallel supports 30 mm apart, and a bi-directional loading force was supplied by electromagnets attached to the centre of the beam. The load was applied at a frequency of 2 Hz until breakage occurred or 10,000 load cycles had been completed.

If less than 50% of the specimens broke during this time, the test was repeated with the load increased by 4%.

<b>Material</b>	<b>Flexural Fatigue Limit MPa</b>	<b>Statistically Similar Groups</b>
QUIXFIL	75.8 (3.4)	*
Z250	75.8 (2.7)	*
SureFil	79.4 (5.3)	*
Tetric Ceram	64.6 (3.7)	*

**Table 8** Results Flexural Fatigue Limit

As shown in the above table, no statistically significant difference was found between the flexural fatigue limits of QUIXFIL, Z250, and SureFil. The material Tetric Ceram, however, was found to have a significantly lower flexural fatigue limit than any of the other three.

### 3.13 Microleakage in Class II Cavities

**Investigator:** Manhardt, University of Munich

**Clinical relevance:** A good seal between the tooth and the restoration is important in order to prevent ingress of fluids and bacteria, which can lead to secondary caries.

**Method:** Extracted human teeth were filled with the material to be tested, using 4 mm thick layers and curing the material for 10 seconds. The restored teeth were then stored in distilled water at 37°C for 48 hours before being subjected to accelerated aging using the following procedure:

- 2000 thermocycles (5/55°C), with 30 seconds in each bath
- 50,000 cycles of mechanical loading in a chewing simulator, using a 6 mm Degusit-antagonist and 50 N load.

After sealing the apexes and mounting the teeth in plastic, the teeth were then soaked in 5% methylene blue at 37°C for one hour to make any leakage visible. The teeth were finally sectioned and the leakage assessed in both the enamel and dentine junctions.

In the case of the restorative/enamel junction, the number of teeth showing no leakage as far as the dentine enamel junction were counted. It was found that the combination QUIXFIL/Xeno III performed at least as well as the combination SureFil/Prime&Bond NT, and there was no significant difference in the degree of leakage of these two systems.

In the case of the junction in dentine, the number of teeth showing zero leakage at the dentine junction were counted, and results are shown in the Table 9.

**Number of teeth showing zero leakage in the dentine junction**

QUIXFIL / Xeno III	SureFil / Prime&Bond NT
8	0

**Table 9**

In this case it is clear that the combination of QUIXFIL / Xeno III performed better than the combination SureFil/Prime&Bond NT .

**3.14 Polishability and Toothbrush Abrasion**

**Investigators:** Dr JP Salomon, Pr J Déjou : Université de la Méditerranée. UFR d'Odontologie de Marseille. Département de Biomatériaux.

**Clinical relevance:** A smooth surface is of importance for aesthetic as well as practical reasons, since a rough surface not only has a poor gloss, but also allows attachment of bacteria and staining to the surface.

The polishability and surface finish of QUIXFIL were investigated by Salomon as follows. Rectangular shaped samples (5mm width, 8mm length, 1mm thick) were used for this study. The three materials (K-0112 batch n° : UK261851, SureFil: shade A2 batch n°: 020320 and Tetric Ceram : shade A2 batch n° : D65134) were light-cured under a Mylar strip covered by a glass slide. A conventional halogen unit (ELIPAR TRILIGHT ; 3M/ESPE) in a standard curing mode (850mW/cm<sup>2</sup>) was used according to the exposure time recommended by the manufacturers (40s). The materials were then finished and polished using either a 30µm diamond burr, and followed by treatment with an one of Occlubrush, Enhance, or Soflex discs.

The mean roughness values (Ra) after each treatment are summarised in Table 10.

Finishing method	Mean Roughness directly after finishing			p =
	Quixfil m(sd)	Surefil m(sd)	Tetric ceram m(sd)	
30 µm	2.54 (0.36) <sup>a 2</sup>	2.83 (0.38) <sup>ab 2</sup>	2.94 (0.75) <sup>b 2</sup>	<b>0.03</b>
30 µm + Occlubrush	2.44 (0.40) <sup>a 2</sup>	2.83 (0.41) <sup>b 2</sup>	2.95 (0.55) <sup>b 2</sup>	<b>0.003</b>
30 µm + Enhance	0.21 (0.05) <sup>b 1</sup>	0.17 (0.04) <sup>ab 1</sup>	0.12 (0.03) <sup>a 1</sup>	<b>&lt; 0.0001</b>
Soflex	0.20 (0.03) <sup>b 1</sup>	0.12 (0.02) <sup>a 1</sup>	0.10 (0.01) <sup>a 1</sup>	<b>&lt; 0.0001</b>
	p = <b>&lt; 0.0001</b>	<b>&lt; 0.0001</b>	<b>&lt; 0.0001</b>	

For each row, groups with the same superscript letter did not significantly differ

For each column, groups with the same superscript figure did not significantly differ

**Table 10**

Although there are small but statistically significant differences between values for the different materials and treatments, all results are satisfactory and differences are clinically insignificant. It can therefore be stated that QUIXFIL can be polished at least as well as either SureFil or Tetric Ceram.

### Roughness after tooth brush abrasion

The second part of the polishing test was an evaluation of the effect of a tooth-brushing simulation on the surface roughness of K-0112, Surefil and Tetric Ceram after finishing/polishing with (1) diamond bur (30µm grit), (2) diamond bur and Occlubrush, or (3) diamond bur and Enhance system. Results are summarised in Table 11.

Finishing method	Mean Roughness after toothbrush abrasion			p =
	Quixfil m(sd)	Surefil m(sd)	Tetric ceram m(sd)	
30 µm	2.82 (0.17) <sup>3</sup>	2.78 (0.27) <sup>3</sup>	2.78 (0.29) <sup>3</sup>	<b>NS</b>
30 µm + Occlubrush	2.29 (0.15) <sup>a 2</sup>	2.51 (0.23) <sup>b 2</sup>	2.31 (0.18) <sup>a 2</sup>	<b>0.004</b>
30 µm + Enhance	0.45 (0.17) <sup>1</sup>	0.39 (0.13) <sup>1</sup>	0.45 (0.21) <sup>1</sup>	<b>NS</b>
	p = <b>&lt; 0.0001</b>	<b>&lt; 0.0001</b>	<b>&lt; 0.0001</b>	

For each row, groups with the same superscript letter did not significantly differ

For each column, groups with the same superscript figure did not significantly differ

**Table 11**

Again, differences were either not statistically significant or significant but small, and in no case was a difference in roughness clinically significant. It can therefore also be stated that the polish of QUIXFIL is retained after toothbrush abrasion at least as well as for SureFil and Tetric Ceram.

## 4 Step-by-Step Instructions

### 1. Preparation

Clean tooth to be prepared with a rubber cap and a prophylactic paste such as Nupro®.

Preparation may be kept to the minimum required for caries removal.

Finishing of cavity margins with a fine finishing bur will contribute to marginal quality.

After preparation, wash cavity thoroughly with air/water spray.

Remove rinsing water by blowing gently with an air syringe or blot-dry with a cotton pellet.

Do not desiccate the dentine structure.

### 2. Pulp Protection

For direct or indirect pulp-capping protect the dentine close to the pulp (< 1mm) with a hard-setting calcium hydroxide liner (e. g. Dycal®), leaving the remaining cavity surface free for bonding with the adhesive.

### 3. Conditioning and application of adhesive

see Figures 14 and 15 for Xeno III Single Step Self-Etching Adhesive.

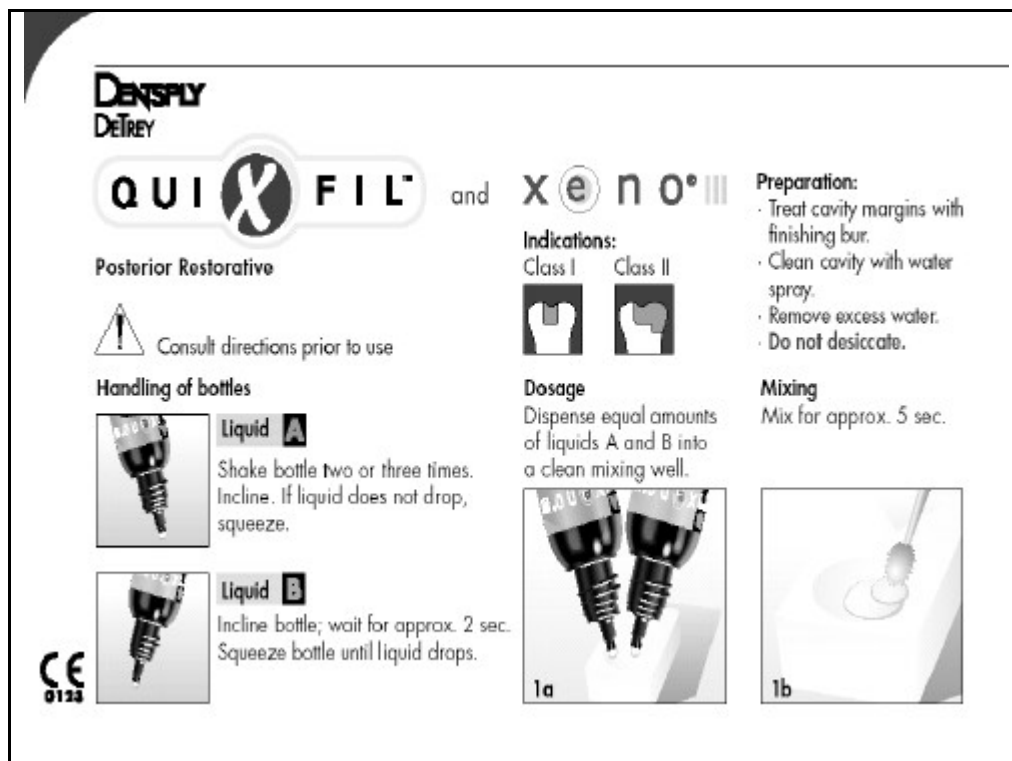
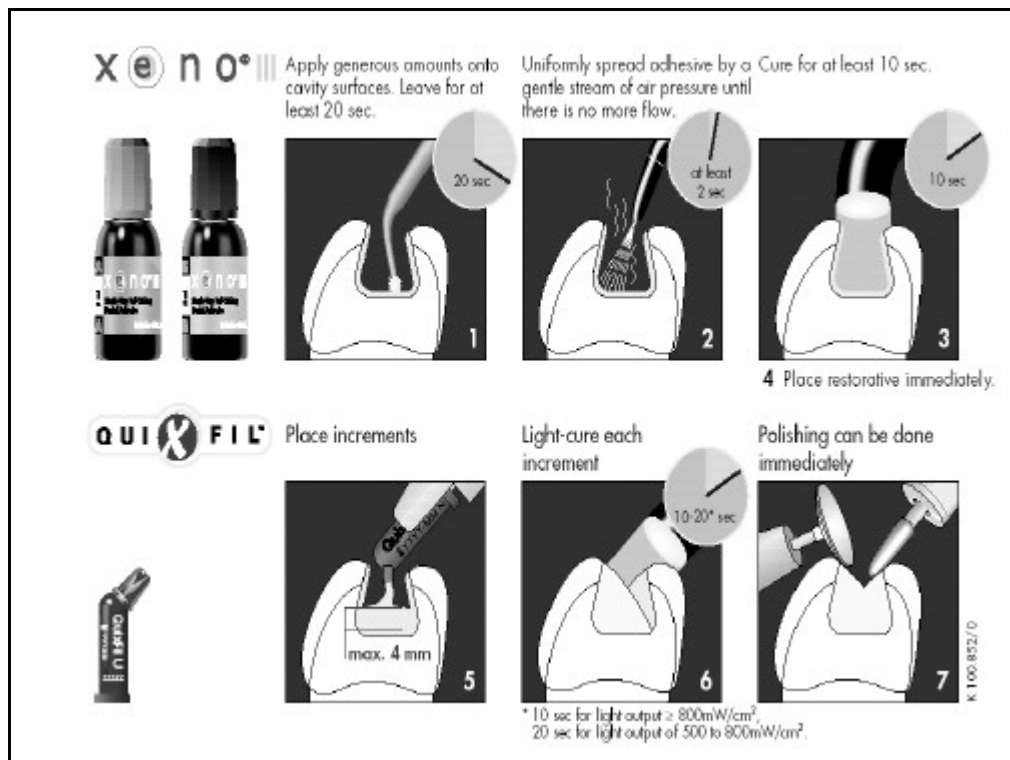


Figure 14



**Figure 15**

#### **4. Placement of QUIXFIL**

Insert Compules tip into the notched opening of the applicator gun barrel.

Dispense QUIXFIL directly into the cavity preparation. Due to the high depth of cure and low shrinkage of QUIXFIL, placement in layers up to 4 mm thick is possible.

#### **5. Curing**

Cure each increment separately with a light curing unit:

20 seconds for units with an output of 500 to 800 mW/cm<sup>2</sup>.

10 seconds for units with an output of 800 mW/cm<sup>2</sup> and more.

The tip of the light guide should be held as close as possible to the restoration during curing.

Important: Be sure to expose each area of the entire restoration to the curing light.

Additionally, the restoration should be cured through lingual or buccal enamel walls.

#### **6. Finishing**

Begin finishing immediately after curing. Gross excess material may be removed with fluted finishing burs or diamonds. Finishing is best achieved by using Enhance™ Finishing and Polishing Discs and interproximal finishing and polishing strips. In patients with an adequate oral hygiene, the final high lustre of the restoration develops with use.

## 5 Summary of Clinical Studies

### 5.1 Clinical Investigation of the restorative system k-0112 (QUIXFIL) and Xeno III for Class I and II restorations of the University of Hong Kong

**Objectives:** Demonstration of the product's safety and efficacy regarding its unrestricted use in posterior teeth for all Class I and II restorations (an alternative for dental amalgam). Criteria to evaluate were pulp and gingival compatibility, marginal quality (sealing properties), retention, surface quality, resistance to occlusal stress and wear, shade match and colour stability.

**Design** Prospective, longitudinal, uncontrolled Clinical Investigation according to Revised (1989) ADA Guidelines for Composite Resin Materials for Posterior Restorations  
(Figures 16-19)

Revised <b>ADA</b> Clinical Protocol Guidelines for submission of Composite Resin Materials for Posterior Restorations (1989)	
Patients and Restorations for Clinical Investigations	
Patients	≥ 30 at baseline ≥ 25 at 2 years ≥ 20 at 4 years
Teeth	First or second molars Must be in occlusion
Cavity Class	≥ 75 % Class II
Cavity Size	≥ 1/3 intercuspal distance
Cavity Type	≥ 10 complex restorations

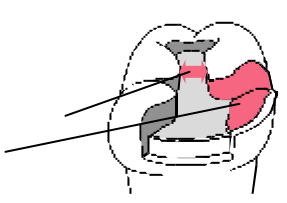


Figure 16

Revised **ADA** clinical Protocol Guidelines  
for submission of Composite Resin Materials  
for Posterior Restorations (1989)

Acceptance Criteria	2 years	4 years
Maintenance of colour	< 10% Charlie	< 10% Charlie
Marginal discoloration	< 10% Charlie	< 15% Charlie
Marginal integrity	< 5% Charlie	< 10% Charlie
Caries - recurrent or marginal	< 5% Charlie	< 10% Charlie
Maintenance of interproximal contact	< 5% observable broadening	< 10% observable broadening

No more than 5% Delta (bulk fracture) at any time.

Figure 17

**ADA** Posterior Composites Acceptance Program

Required Wear Resistance

Wear Measurement	Maximum Allowed Wear (MW)			
	Restricted Category		Unrestricted Category	
	6M-2Y	6M-4Y	6M-2Y	6M-4Y
Average for restoration	125	200	75	150
Local (occlusal contact)	400			

Figure 18

<b>ADA</b> Posterior Composites Acceptance Program Cumulative Failures: Marginal Integrity Failures + Caries + Wear Failures + Replacements		
Indication	Maximum Allowed Cumulative Failures	
	at 2 years	at 4 years
Restricted Use	8 %	15 %
Unrestricted Use	5 %	10 %

**Figure 19**

**Modifications with Regard to ADA** Higher number of patients and restorations.

**Guidelines:**

**Investigator/s**

Dr. Gary S.P. Cheung, Dr. Edward Lo.

**Number of Patients**

30 at recalls.

(K-112 only)

**Number of Restorations**

30 at recalls

(K-112 only)

**Acid Conditioner/s**

None, as a self conditioning adhesive was used

**Adhesive/s**

Xeno III, single step self-etching dental adhesive

**Method of Evaluation**

Clinical examination, rating according to Cvar and Ryge.  
 Indirect evaluation of selected cases for wear.

**Recall Periods**

Baseline, **3-**, **6-** and 12, 24, 48 month

(those reported on are printed in bold)

**Success Criteria**

According to ADA Acceptance Criteria, with the understanding that for the 3, 6 and 12-month recalls the incidence of failures may at the most be half of what is accepted for the 24-month recall.

## Summary of 3-Mth Results

45 restorations (11 Class I, 31 Class II, and 3 complex) were placed in 36 patients.


In the Preliminary Report of December 6, 2002, data on 30 restorations in 30 patients were provided. In comparison to baseline, no changes in restorative quality (colour match, marginal discolouration, marginal integrity, recurrent caries, anatomic form, surface texture, swelling out, gingival status) were reported. Survival rate at reporting date was 100%.

In the Summary Data Sheet provided on 19 March 2003, data on 32 restorations were provided. One failure (3%) was reported on (Tooth with pre-operative signs of pulp irritation (replacement due to sensitivity especially to cold)).

## 6-Month Results

(Table 12)

In the 6-month Report provided on 19 March 2003, data on 35 restorations were provided. The failure rate of 3% remained unchanged.

		<b>Clinical Investigation Class I, II Hong Kong 6-mth Data</b>			
		Percentage of Scores (%)			
Ryge (USPHS) Criteria		Alpha	Bravo	Charly	Delta/ Oscar
• Margin integrity		100	0	0	0
• Margin discolouration		100	0	0	n.a.
• Recurrent caries		100	0	n.a.	n.a.
• Anatomic form		100	0	0	n.a.
• Surface texture		100	0	0	n.a.
• Colour match		68	29	3	n.a.
• Sensitivity		97	3	n.a.	n.a.

Interim Report 2003-03-24 by G.S.P. Cheung on 35 K-0112 (QuixFil) restorations (n=35 for sensitivity, for all other criteria n=37)

**Table 12**

**Conclusions by the investigator** The results of this clinical investigation demonstrate at this

stage the safety and utility of QUIXFIL for the intended indications. The results indicate a level of performance as required for the ADA Acceptance Program.

## **5.2 Clinical Investigation of the restorative system K-0112 (QUIXFIL) and Xeno III for Class I and II restorations at the University of Munich**


**Objectives:** Demonstration of the product's safety and efficacy regarding is unrestricted use in posterior teeth for all Class I and II restorations (an alternative for dental amalgam). Criteria to evaluate were pulp and gingival compatibility, marginal quality (sealing properties), retention, surface quality, resistance to occlusal stress and wear, shade match and colour stability.

<b>Design</b> (see Figures 16-19)	Prospective, longitudinal, controlled Clinical Investigation according to Revised (1989) ADA Guidelines for Composite Resin Materials for Posterior Restorations.
<b>Modifications with Regard to ADA Guidelines:</b>	Higher number of patients and restorations. Inclusion of a reference material.
<b>Investigator/s</b>	Prof. Dr. Reinhard Hickel, Dr. Jürgen Manhart, Dr. Lidka-Karin Thiele, Dr. Petra Neuerer
<b>Number of Patients</b>	40 at recalls.
<b>Number of Restorations</b>	40 at recalls
<b>Acid Conditioner/s</b>	None, as a self conditioning adhesive was used
<b>Adhesive/s</b>	Xeno III, single step self-etching dental adhesive
<b>Control Material/s</b>	Tetric ceram, Syntac classic
<b>Method of Evaluation</b>	Clinical examination, rating according to Cvar and Ryge, indirect evaluation of selected cases for wear.
<b>Recall Periods</b> (those reported on are printed in bold)	Baseline, 3-, <b>6-</b> and 12, 24, 48 month
<b>Success Criteria</b>	According to ADA Acceptance Criteria, with the understanding that for the 3, 6 and 12-month recalls the incidence of failures may at the most be half of what is accepted for the 24-month recall.

## 6-Month Results

(Table 13)

In the Preliminary 6-Month Report of March 17, 2003, Manhart reports that 46 restorations in 30 patients were in situ and functional. There was no statistically significant difference in the performance of the test material and the control (a conventional fine-particle hybrid composite placed in combination with a multi-bottle bonding system and phosphoric acid conditioning).

				
Clinical Investigation Class I, II Munich 6-mth Data				
Percentage of Scores (%)				
Ryge (USPHS) Criteria	Alpha	Bravo	Charly	Delta/ Oscar
• Margin integrity	0%	0%	0%	n.a.
• Margin discolouration	97%	3%	0%	n.a.
• Recurrent caries	100%	0%	n.a.	n.a.
• Anatomic form	100%	0%	0%	n.a.
• Surface texture	97%	3%	0%	n.a.
• Colour match	100	0	0	0

Interim 6-mth Report by J. Manhart 2003-03-17 on 35 K-0112 (QuixFil) restorations

**Table 13**

### Conclusions by the investigator

The recall examination of restorations being in situ for 6 month showed in the majority of cases excellent (alpha ratings) clinical results.

## 6 References

1. Feilzer AJ, de Gee AJ, Davidson CL, Curing contraction of composites and glass-ionomer cements, *J. Prosthet. Dent* 59:297-300 (1988)
2. McConnell RJ, Johnson LN, Corazza L, Gratton DR, Dimension change during setting of composite resin, *J. Dent Res.* 73:SI;126/196
3. Feilzer AJ, Afrodite I, de Gee AJ, Davidson CL, The influence of water sorption on the development of setting shrinkage stress in traditional and resin-modified glass-ionomer cements, *Dent Mater* 11:186-190, (1995)
4. Soltész U, Leupolz M, Dimensionsverhalten von Glasionomerzementen in trockener und feuchter Umgebung, *Dtsch Zahnärztl Z* 48, 431-435 (1993)
5. Attin T, Buchalla W, Keilbassa AM, Hellwig E, Curing shrinkage and volumetric changes of resin-modified glass-ionomer restorative materials, *Dent. Mater* 11:359-362, (1995)
6. E. A. Fogleman et al., *Dental Materials* 18 (2002) 324-330
7. Martin N, Jedyakiewicz NM, Measurement of Hygroscopic expansion of composite restoratives, *J. Dent Res.* 74:SI;462/493 (1995)
8. P. Bath, U. Soltész, B. Klaiber, Polymerisationschumpfung, Wasseraufnahme und Quellung von ausgewählten Komposit Füllungsmaterialien. Report W2/85 from Fraunhofer-Institute Für Wekstoffmechanik
9. de Gee AJ, Pallav P., Occlusal wear simulation with the ACTA wear machine, *J Dent* 1994;22 Suppl 1:S21-7
10. Hara AT et al., *Am J Dent* 2001 Dec; 14(6):383-6
11. Leinfelder KF, Suzuki S., In vitro wear device for determining posterior composite wear *J Am Dent Assoc* 1999 Sep;130(9):1347-53