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Title: Determination of Surface and Subsurface Fatigue Damage in Dental Composites

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Abstract: Abstract

Dental filling materials are subjected to cyclic compression in the mouth. Nine resin-based composite filling materials were subjected to 2000 compression cycles between either 0 and 12 kg, or 0 and 40 kg. Surface deformation was measured as the diameter of the compression scar and surface microhardness determined by a Vickers' microhardness test at 4 sites around the scar. Subsurface damage was stained with silver nitrate and the area of stain determined by image analysis software. Subsurface microhardness was measured at 4 sites around the stained zone. Surface deformation at 12 kg was inversely proportional to the surface microhardness at 12 kg. At both loads subsurface damage was directly proportional to the subsurface microhardness. Samples with small filler particles experienced less subsurface damage than those with larger particles. Silver nitrate staining was found to be a useful method for identifying subsurface damage.

Novelty Statement

We confirm that this paper is an original paper that has not been previously, nor simultaneously, in whole or in part, submitted anywhere else.

Determination of Surface and Subsurface Fatigue Damage in Dental Composites

Introduction

Teeth and dental restorations are subject to cyclic loading during both eating and at other times. Human beings often clench or grind their teeth together at times of mental concentration or stress. The latter often occurs at night and is known as bruxism. The forces applied to the natural teeth while eating may vary between 0.5 and 25 kg [1-3]; but during nocturnal grinding these forces may reach 80 kg [4]. Besides causing of abrasion of the surface these forces can also initiate cracks in the subsurface [5,6]. Today, dental resin composite materials are commonly used to replace missing tooth tissue that has been worn away by grinding [7-9]. They have the advantage that they can be directly stuck onto the surface without requiring physical locking keyways to be drilled into the teeth [10].

Dental resin composite consist of glass filler particles embedded in a polymer matrix based on an epoxy resin (Bisphenol A glycidyl methacrylate). The functions of the fillers are to reduce the polymerization shrinkage on setting and to increase wear resistance. The first composites were introduced in the mid 1960's and advocated solely for fillings in the front teeth that are not subject to heavy chewing forces. The fillers were relatively large quartz particles >50 μm in diameter. By the mid 1980's a new generation of materials became available for use in the back teeth that are subject to the forces of grinding and clenching. These materials required radiopaque glass fillers to make them visible on radiographs. The particle size had decreased to below 10 μm ; that was still relatively large compared to modern materials. Between 1985 and 1990 small amounts of colloidal silica (20 nm dia) were added to "fill in" gaps between the glass particles. Materials containing glass particles and colloidal silica were called hybrid composites. Between 1990 and 1995 the size of the glass particles was reduced to around 1 μm in diameter. Again these materials contained some colloidal silica. They were called microhybrid materials and the majority of dental composites currently available are of this type. Another type of composite is based entirely on colloidal silica that has been agglomerated into pre-polymerised

particles during the manufacturing process. The materials are called microfilled composites.

Dentists place composite fillings by pressing the material into the cavity with small packing instruments. The packing force bring the resin to the surface so that the surface layer tends to resin rich compared to the subsurface. Also, the surface layer does not set as hard as the underlying material because free radicals generated from the initiator system compete with oxygen in the atmosphere giving rise to an oxygen inhibited layer on the surface [11, 12].

In 1981 Wu and Cobb developed a method to stain subsurface damage in resin composites with silver nitrate [13, 14]. Silver ions (Ag^+) penetrate through the network of cracks and can subsequently be precipitated as colloidal ionic silver (Ag) in the damaged zone. The color of the stained zones results from the scattering of light by these colloidal silver particles according to Mie Theory for the scattering of light by colloidal particles [15]. The purpose of this study was to determine the area of surface deformation and subsurface damage in nine dental resin composites caused by low cyclic fatigue. The values were related to the composition and properties of the materials.

Materials and Methods

Nine dental resin composite filling materials were used in the investigation. The materials are described in Table 1. For the purpose of statistical analysis the materials are categorized as having Large, Medium or Micro particles. Figure 1 shows micrographs of the 9 materials to illustrate the particle size and configuration.

Twenty disks of each of the materials were fabricated by condensing them into a PTFE mould (diameter 7mm x 2.5mm). Seven of the materials were set by photo-initiation whilst the remaining two were supplied as a two paste system and set by chemical initiation after mixing. The specimens were subjected to 2000 compression cycles in a Universal Testing Machine (Nene Instrument, Wellingborough, UK) using a rounded cone indenter. Ten specimens were cycled between 0 and 12 kg and the remaining 10 disks between 0 and 40 kg. All cycling

was performed under water. A schematic of the apparatus is shown in Figure 2. The testing machine recorded the increasing displacement of the indenter during the test.

To determine the degree of surface damage the diameter of the compression scar was measured with an eyepiece graticule, using an incident light microscope (Photomicroscope II, Carl Zeiss, Oberkoken, Germany) with Differential Interference Contrast (DIC) to enhance the boundary of the scar. Surface microhardness was determined by making four Vickers' indentations around the scar using a commercial microhardness tester (MPH, Carl Zeiss, Oberkoken, Germany). The indentations were made with a load of 200 grams that was allowed to stabilize for 30 seconds before measurement. This is standard test for dental composites. The position of the surface scar and indentations is shown in Figure 3A.

To determine the extent of subsurface damage after compression, the specimens were immersed in AgNO_3 (3 mol/L) for 72 hours. The specimens were then sectioned through the indentation scar with a diamond cutting disk (Isomet, Buehler UK, Coventry, UK.) to expose the subsurface. The sectioned specimens were then embedded in PMMA and ground on silica carbide papers (240, 400, 600, and 1200 grit) to achieve a flat surface. Finally, they were polished on microcloth with 50 μm alumina in an ultrasonic polishing machine (Vibromet Polisher, Buehler UK, Coventry, UK).

The area of stained subsurface damage was examined with an the incident light microscope using crossed polars to exclude directly reflected light and reveal the color of the stain. Digital images were taken and the damaged zoned measured using image analysis software (Sigma Scan Pro 5.0, SPSS, USA). The microhardness of the composite surrounding the damage zone was measured in four areas with the microhardness tester. The position of the stained zone and subsurface indentations is shown in Figure 3B.

The values for the diameter of the surface scar and the area of subsurface damage were analyzed by one-way Analysis of Variance (ANOVA) followed by

Scheffe *post hoc* tests to determine homogeneous subsets (level $p = 0.05$). Correlations between the four values: surface deformation, surface microhardness, subsurface damage and subsurface microhardness, for the individual specimens, were made by Pearson's Correlation coefficient. Correlations between the categorized size of the particles and the degree of surface deformation and subsurface damage were determined by Kendall's Coefficient (tau-c).

Results:

Surface deformation

The values for the diameter of the deformation scar and surface microhardness are given in Table 2. Table 3 shows the homogeneous subsets for surface deformation.

These results shows that for all materials the size of the scar increased with the increased load. The increase in size was not directly proportional to the increased load.

Subsurface damage

The values for the area of subsurface damage and subsurface microhardness are given in Table 4 and the homogeneous subsets are shown in Table 5.

In terms of subsurface damage, at 12 kg there were four subsets with three of materials showing no apparent damage. At 40 kg all the materials except MiH3 displayed some damage. The post-hoc test discriminated 5 subsets.

Figure 4 shows representative examples of the subsets indicated in Table 5. The depression caused by surface deformation can be seen on the samples at 40 kg; but not at 12kg.

Figure 5 shows a Scanning Electron Micrograph (x2000) of MPH1. Although there are signs of debonding of the resin from the larger particles (arrowed) the damaged zone is not as well demonstrated as with silver nitrate staining.

Statistical correlations

The correlations between for the experimental values of the individual samples are given in Table 6

The correlation values (τ -c) between the categorized filler particle size and the ranked deformation, damage and hardness values are given in Table 7.

The regression analysis indicated a significant inverse relationship between the diameter of the deformation scar at 12Kg values ($r=-0.51$, $p<0.001$); but the value was only just significant at 40Kg ($r=-0.34$ $p=0.04$). In terms of subsurface damage there was a significant correlation between the degree of subsurface damage and the microhardness of the materials. This was stronger at 40 kg than at 12 kg.

Discussion :

In this study silver nitrate was used to stain the zone of subsurface damage. It has been shown that this technique can identify damaged zones that are not apparent with scanning electron microscopy. The reason for this is that silver nitrate is not a stain in the traditional sense of the word. The latter are essentially dyes with large complex molecules that cannot penetrate into subsurface crazes and cracks. Silver nitrate is a colourless liquid that penetrates as the silver ion (Ag^+). It is only after the silver has been reduced to ionic silver in the cracks and crazes that the colour becomes apparent because of light scattering by these particles that are 5-30nm in diameter [15].

Areas of silver staining similar to that show in Fig. 4 have been demonstrated in actual fillings that had been removed for replacement after a number of years in the mouth [16]. There was often a wear scar on the surface of the filling adjacent to the subsurface stain.

It could be argued that the damage is not cumulative; but resulted from the initial "impact" of the indenter. This is unlikely because the depth (and therefore the volume) of the indentation increased over the 2000 cycles.

The results indicated that after 2000 compression cycles at 12 kg there was a significant correlation between surface deformation and surface hardness. The fact this correlation was much weaker at 40 kg can be explained because, as discussed in the introduction, the surface of a dental composite is softer than the bulk of the material because of the resin rich effect and oxygen inhibition of the free radicals during conversion. As the overall depth of penetration at 12 kg was less than at 40 kg the penetration of the indenter is more likely to be influenced by the surface rather than the subsurface hardness. The deeper the penetration the more the process is influenced by the subsurface hardness.

With the exception of MPH1, there was no significant difference between the surface hardness values at the two loads and for some materials the mean values at 40Kg was less than at 12kg (Table 3). The differences are just minor differences in surface softness that can be caused by the packing pressure of the composite into the mould as this influence the depth of the resin rich layer. The large mean value for MPH1 resulted from a very high reading for one specimen. This can be caused if the Vickers' microhardness indenter pressed directly on a filler particle.

For all materials the degree of subsurface damage increased with the increased load. For some materials there was almost 4 times the degree of damage at the higher load. At 12 Kg there was no evidence of subsurface damage with three materials based on microparticles. These materials were also in the lowest subset for damage at 40 kg. The three large particle composites were all in the largest subset at 40 kg. Again, there was no significant difference between the subsurface hardness values between 12kg and 40 kg (Table 4). This militates against the concept that hardness may increase because the particles are compressed by flow of the materials during deformation.

Table 7 shows a high degree of correlation between the categorized particle size and both the microhardness, surface deformation (at 12 Kg) and subsurface damage. The hardness of the materials must be related to the filler size and packing rather than the hardness of the particles because the glass in both some

of the medium sized particle and micro particle composites is the same. The statistical relationship was only significant if the materials were categorized into large, medium and microfilled materials and correlated with the ranked values for deformation and damage. The correlation between the values and the actual size of the particles for the 9 materials was not significant. The fact that the microfilled materials showed comparatively little damage, as identified by stain, may be because the materials can deform elastically rather than nucleating fatigue cracks.

Today clinicians use composites to cover the biting surface of worn teeth [17,18]. Clinical studies with the early dental resin composites in the 1970's showed excessive wear if these were used on the biting surface of the back teeth. These early materials had large filler particles. By 1985 the fillers were in the medium range. The results of a 10-year clinical trial indicated that these materials had adequate wear resistance for the back teeth[19]. More recent studies have shown that the new microhybrid materials also have an adequate wear resistance in the mouth [20,21].

Conclusion:

Surface Deformation and Subsurface damage varied between different resin composites. Overall, the softer the material surface, the more the surface deformation. In contrast, the harder the subsurface, the more the subsurface damage.

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Figure 1
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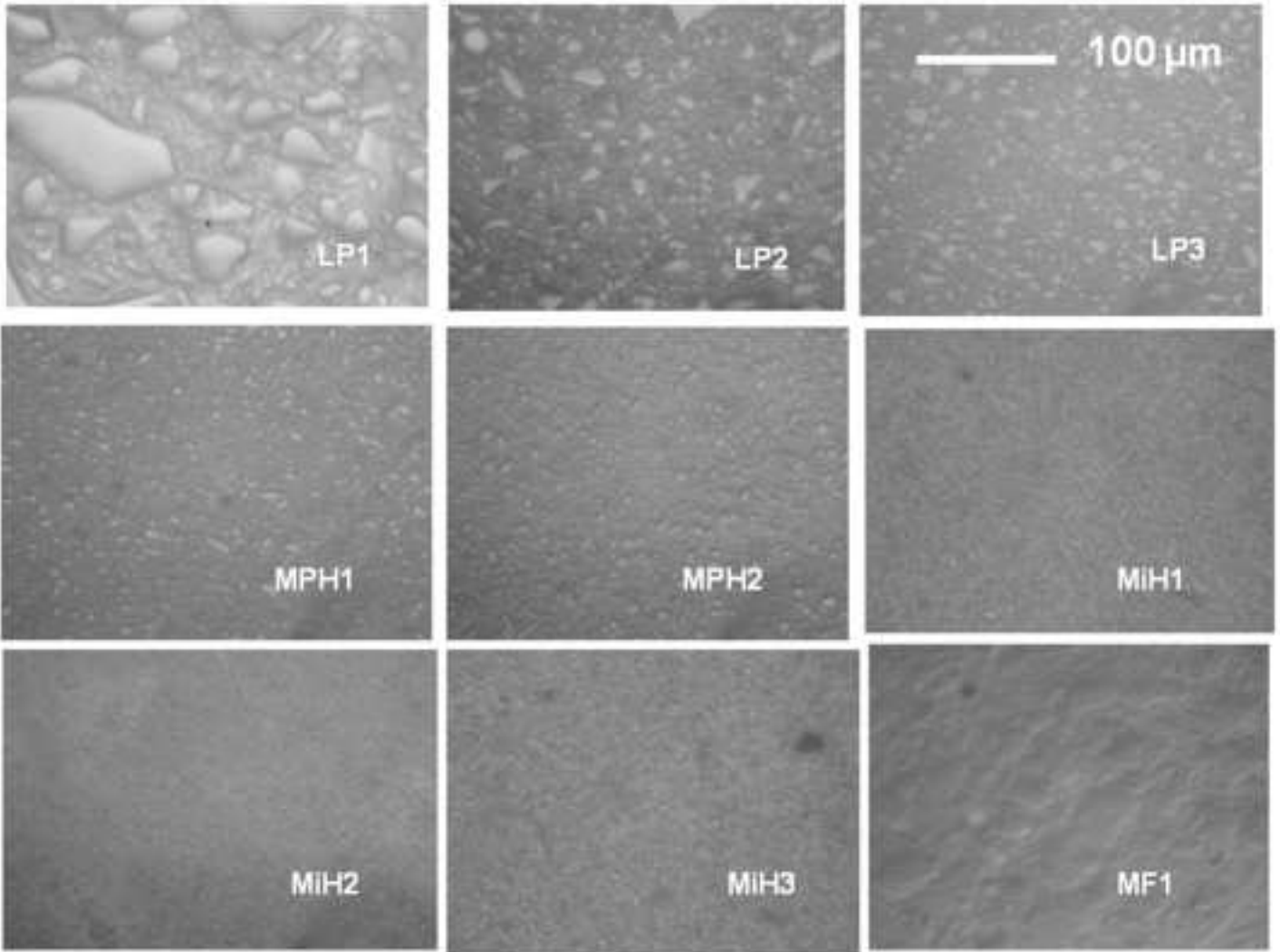


Figure 2
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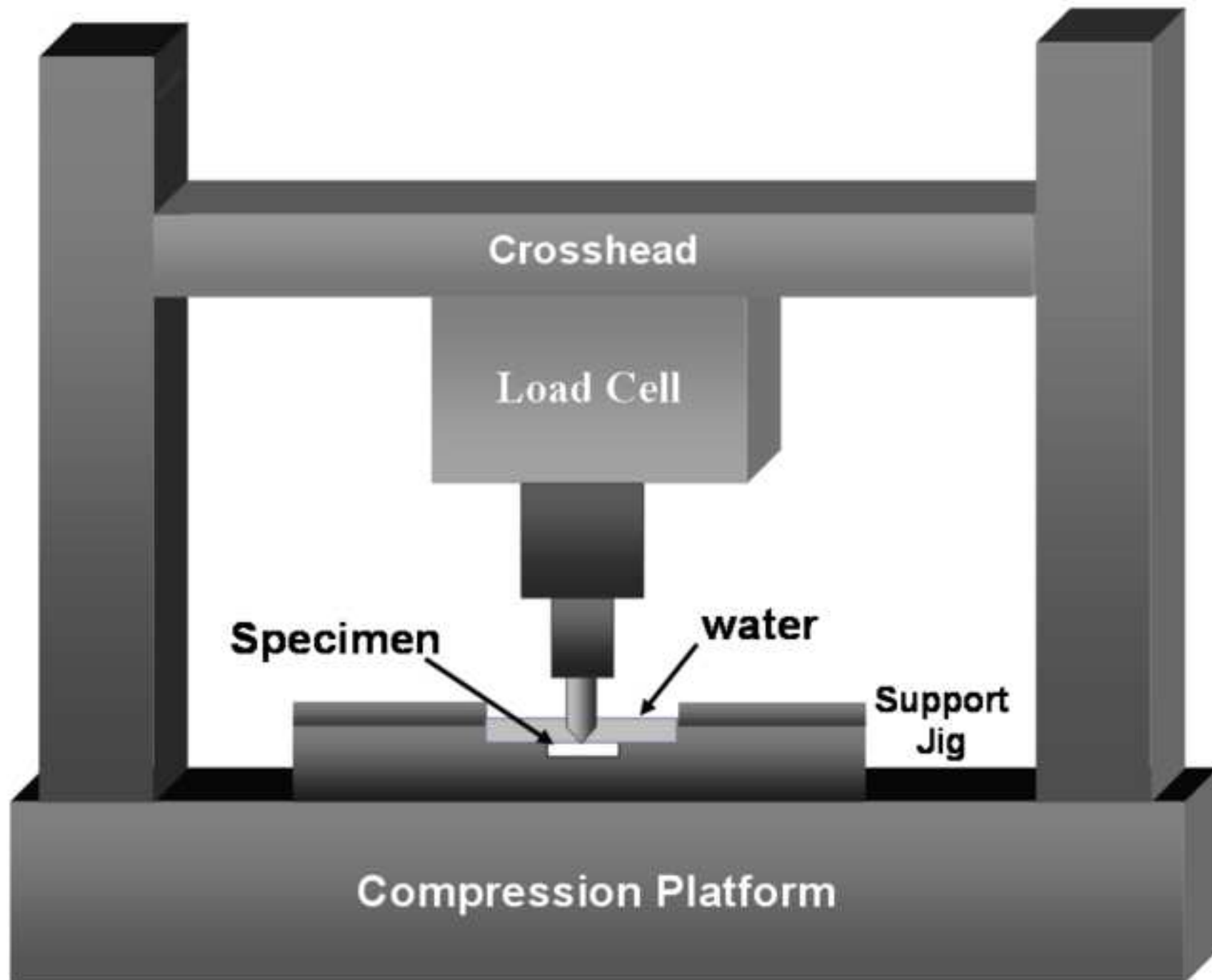


Figure 3
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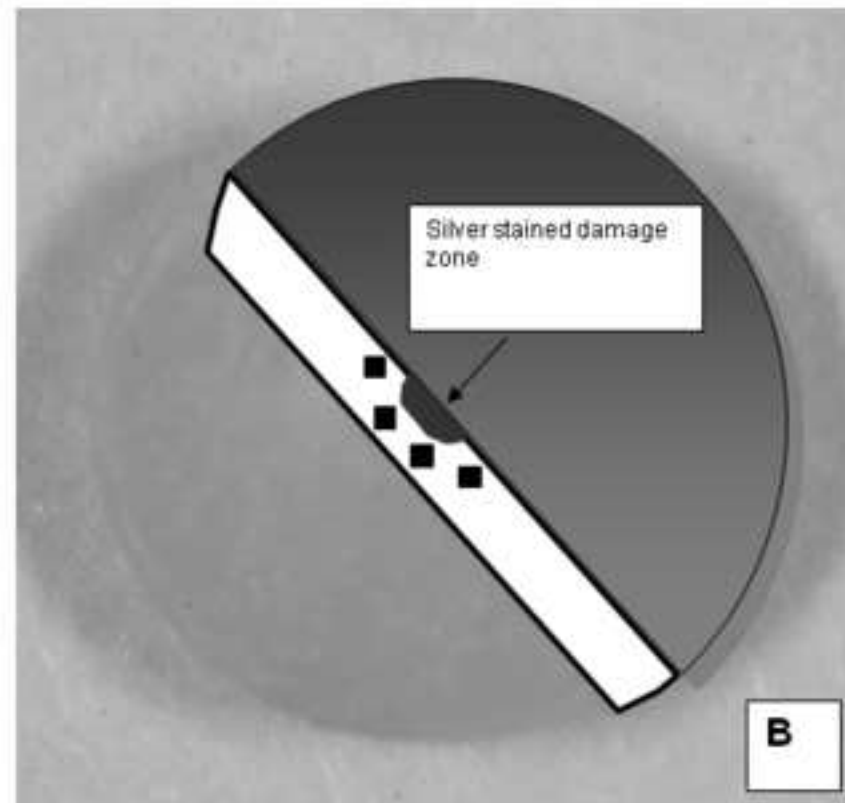
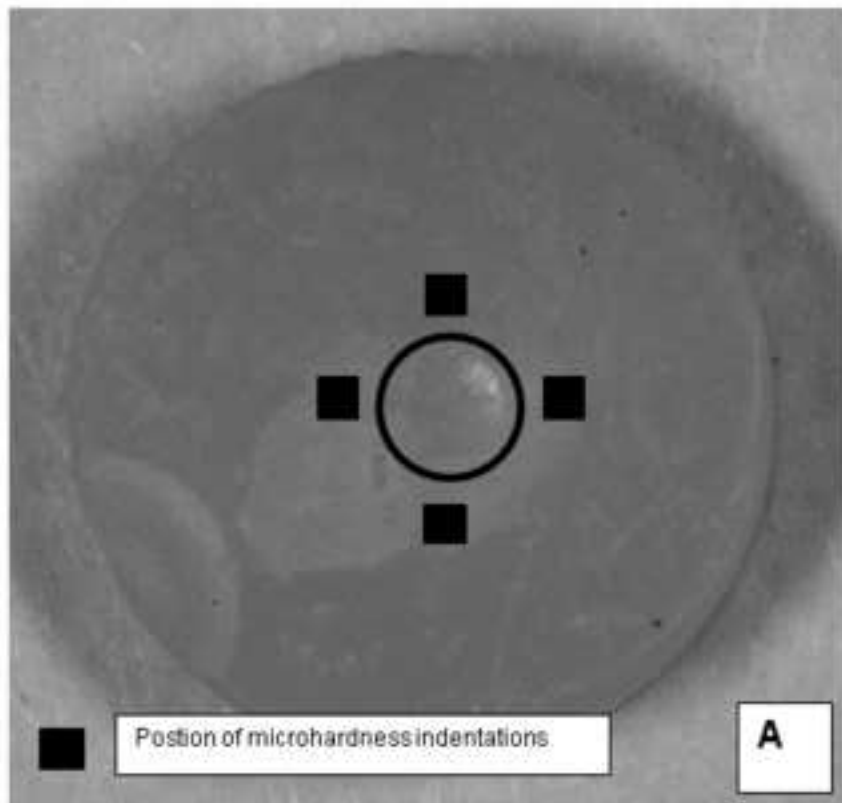


Figure 4

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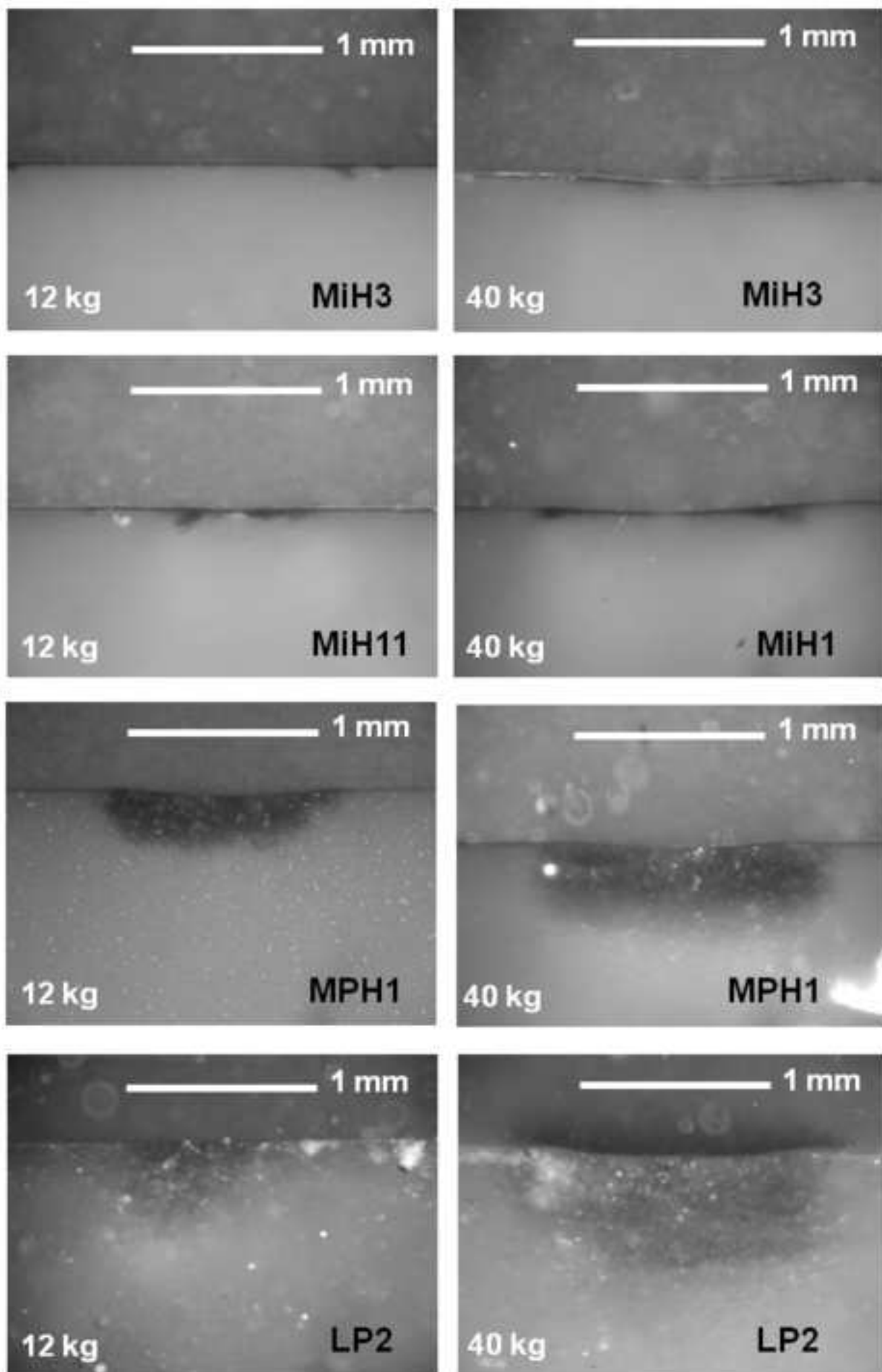
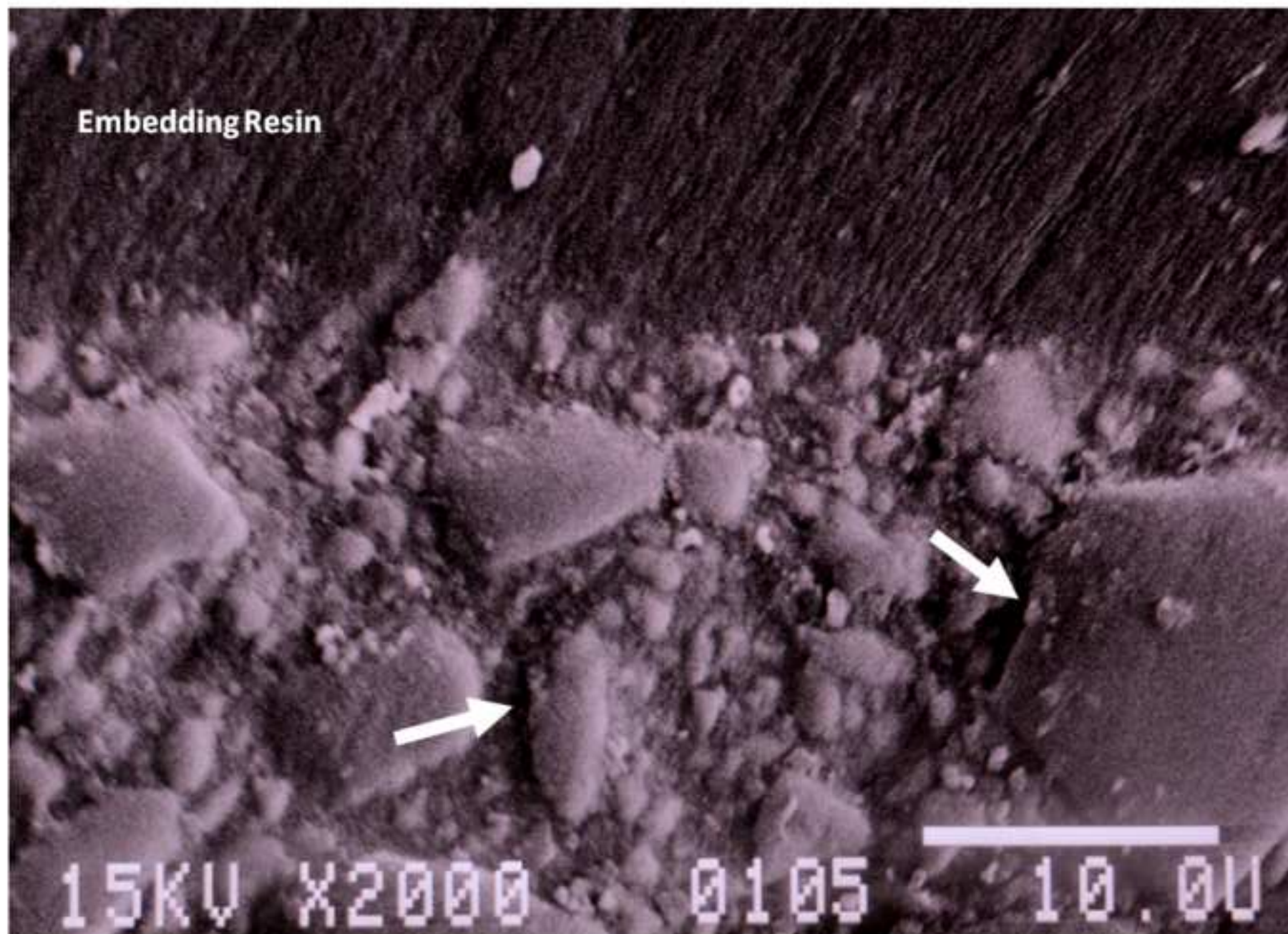


Figure 5
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Responses to Referees

The referees comments were very helpful. We had written the paper primarily to introduce the technique of silver nitrate staining as a means to identifying damage in dental composites that cannot be identified by electron microscopy. Both referees commented that the paper was interesting. The referees correctly pointed out that we had overlooked some of the basic science. There is a danger that in making the revisions the original reason for submission would be lost. Nonetheless, we have altered the paper considerably in the light of the comments. It is clear from both referees comments that we did not give enough detail about the technique and , hopefully, this has been rectified. It is helpful to consider the second referees comments first.

Referee 2.

1. We have removed all the commercial names from the manuscript. However, whilst we hope that the majority of readers will not be dentists, the paper may be accessed by a few dental researchers. They would expect to see commercial names and batch numbers to compare with papers in dental journals. We have put these at the back of the paper. The editor can, of course, leave them out at his discretion.

2. There was no change in microhardness are a result of deformation. Table 2 & 4 show that the microhardness was not significantly different between the samples that had been cycled at 12 kg and the ones cycles at 40kg. The differences are just non - significant differences between samples. There was a difference between the surface hardness and the subsurface hardness of the materials. This is because of the surface resin rich layer and the oxygen inhibition of the setting reaction at the surface. This has been more clearly explained in the text.

3. The text has been changed throughout to “low cycle fatigue”

4. Silver nitrate staining was the focus of my PhD (1990) and I have published a number of papers on this subject. The area of damage does increase with increasing cycles. In the present experiment the universal testing machine provided a screen display of overall displacement of the indenter showing that the depth of indentation increased with increasing cycles. Given its geometry (rounded cone) the increase in depth must be accompanied by an increase in volume. This has been included in the text.

5. The cited papers: L.H. Mair, The colors of silver with silver nitrate staining in dental materials, Dent. Mater. 8 (1992) 110-7. and L.H. Mair, Subsurface compression fatigue in seven dental composites, Dent. Mater. 10 (1994) 111-115 shows a number of SEM micrographs of the area stained by silver nitrate. There is normally no evidence of damage on the SEM which is why the silver technique is useful. We have now included a SEM for this paper. We have reemphasised that silver nitrate is not a dye in the traditional sense of the word. It penetrates into the damaged zone as ionic silver (Ag^+). When it is reduced to metallic silver it forms colloidal particles in nano spaces in the material. The brown colour results from the scattering of light by these particles according to Mie Theory. It is not like black and white film where the grains are often microns in diameter. We have emphasised this and included the reference on the colours of silver.

6. I regret that I don't understand the Hertz equation; but having looked it up I think that we would need to use a much more standard material to test this.

7. We have removed the diagrams and replaced them with a more detailed statistical analysis (Kendall's tau-c).

Referee 1

1. This was a most helpful comment. We have changed Table 1 to give more information about the materials and included a statistical analysis of the results in relation to the size of the filler particles. A small point of misunderstanding in comments 5 and 7. This is not an etching technique. Etching means treating the material to open up pre-existing flaws. This is a staining or doping technique. As stated in Comment 5 above – it isn't like using a dye because the AgNO_3 penetrates as an ion rather than macromolecules like most dyes.

2. see 1 above

3. The load and time for the microhardness tests have now been included in text. It is true that the actual zone of damage may be larger than the stained zone. However, there is no way to show this. As stated under the response to Referee 1 (No 5) the silver staining technique indicated damage does not show on SEM. It is not possible to use TEM with these materials because the fillers do not cut cleanly in an ultramicrotome. As composite are two phase materials there is a limit to the magnification that is possible with SEM. The SEM included for this paper is x2000.

4. In this experiment we stained the specimens before sectioning; but in other studies we have stained them after sectioning so that the silver nitrate can penetrate directly into the damaged zone. There is no difference at all in the area of staining detected.

5. Whilst we agree with this comment the resin rich layer has been known about for many years; but as yet, nobody has been able to quantify it. The depth of the resin rich layer is determined by the packing pressure when placing the composite as this brings the resin to the surface. This is not controlled in dentistry because it depends on the shape of filling. It changes from specimen to specimen. We have included the relevant references.

6. The original statistical analysis has been checked and the probabilities are as stated. In response to Comment 1 (above) the data was also correlated to the categorised particle size (Large, Medium, Micro) and, as the referee suggested, there was a highly significant relationship.

7. As stated in comment 3 we cannot demonstrate the presence of cracks because they do normally show on SEM. However, the images in Fig 6 clearly show that something has happened. Whether there are cracks or crazes depend on the definition of crazes. Kambour used silver nitrate to dope crazes in polycarbonate (Polymer 5 (1963) 143-155). This paper shows that AgNO_3 can penetrate these pre-cracks.

I hope that these responses are helpful.

Lawrence Mair

Table 1: Dental resin composites used in this experiment

Material	Code	C A T	Fillers	Mean filler size (μm)	Filler Loading	Surface Hardness (Vickers) (Kg/mm^2)	Subsurface Hardness (Vickers) (Kg/mm^2)
Large particle composite 1	LP1	3	Quartz	80	68.2 vol% 84.0 wt%	101	139
Large particle composite 2	LP2	3	Quartz	50	61.1 vol% 79.7 wt%	67	396
Large particle composite 3	LP3	3	Barium Glass	10	75.5 vol% 85.7 wt%	77	105
Medium particle hybrid composite 1	MPH1	2	Ba-Al-Borosilicate glass particles + Colloidal silica	2.5 μm 20-60 nm	71.4 vol% 87.0 wt%	90.4	116
Medium particle hybrid composite 1	MPH2	2	Ba-Al-Borosilicate glass particles + Colloidal silica	2.5 μm 20-60 nm	65.6 vol% 80.0 wt%	56	103
Micro particle composite 1	MiH1	1	Ba-Al-Borosilicate glass particles + Colloidal silica	1 μm 20-60 nm	57.0 vol% 77.0 wt%	58	98
Micro particle composite 2	MiH2	1	Ba-Al-Borosilicate glass particles+ Colloidal silica	0.7 μm 20-60 nm	56.0 vol% 78.0 wt%	59.6	65
Micro particle composite 3	MiH3	1	Ba-Al-Borosilicate glass particles+ Colloidal silica	0.6 μm 20-60 nm	59.0 vol% 78.4 wt%	62.7	76
Microfilled composite 1	MF1	1	Colloidal silica agglomerated as pre-polymerized particles (PPP)	20-60 μm PPP =30 (μm)	50 vol% 60 wt%	26.2	31

LP = Large Particle

MPH = Medium Particle Hybrid

MiH = Micro Particle Hybrid

MF = Microfilled

Table 2: Results for the surface deformation diameter and surface hardness

Material	Surface Deformation 12 kg (μm)	Surface Microhardness (Kg/mm ²)	Surface Deformation 40 kg (μm)	Surface Microhardness (Kg/mm ²)
LP1	564.0 \pm 43.4	96.8 \pm 19.8	936.0 \pm 72.3	106.6 \pm 6.6
LP2	642.0 \pm 200.4	73.1 \pm 6.2	1176.1 \pm 109	61.0 \pm 4.6
LP3	662.0 \pm 37.0	67.6 \pm 3.1	960.0 \pm 54.8	77.4 \pm 10.0
MPH1	614.0 \pm 115.5	82.8 \pm 3.4	820.0 \pm 21.2	136.45 \pm 78.2
MPH2	722.0 \pm 71.9	55.0 \pm 3.6	910.4 \pm 11.5	56.7 \pm 1.9
MiH1	748.0 \pm 98.8	61.2 \pm 0.2	924 \pm 23.0	55.2 \pm 1.8
MiH2	852.0 \pm 157.9	58.1 \pm 1.7	964.0 \pm 20.7	61.1 \pm 5.0
MiH3	552.0 \pm 31.1	67.9 \pm 6.6	952.3 \pm 24.9	62.7 \pm 0.7
MF1	774.0 \pm 45.1	31.3 \pm 11.2	998.0 \pm 14.8	26.2 \pm 0.9

Table 3: Homogeneous subsets for the surface deformation at 12 and 40 Kg

Subset	Deformation at 12 kg ANOVA F = 4.56, p<0.001								
1	MiH3	LP1	MPH1	LP2	LP3	MPH2	MiH1	MF1	
2			MPH1	LP2	LP3	MPH2	MiH1	MF1	MiH2
Subset	Deformation at 40 kg ANOVA F = 17.48, p<0.001								
1	MPH1	MPH2	MiH1	LP1	MiH3				
2		MPH2	MiH1	LP2	MiH3	LP3	MiH2	MF1I	
3									LP2

Subsets determined by Scheffe Multiple Comparison Test (Level p= 0.05)

Table 4: Results for the subsurface damage and subsurface hardness

Material	Subsurface Damage 12 kg ($\mu\text{m}^2 \times 10^{-3}$)	Subsurface Microhardness (K/mm ²)	Subsurface Damage 40 kg ($\mu\text{m}^2 \times 10^{-3}$)	Subsurface Microhardness (K/mm ²)
LP1	58.9 ± 43.9	139.9 ± 23.7	422.1 ± 81.4	139.0 ± 12.1
LP2	224.6 ± 115.3	454.0 ± 66.7	834.3 ± 112.2	337.1 ± 87.7
LP3	150.0 ± 20.2	105.0 ± 10.2	707.4 ± 108.7	104.6 ± 14.6
MPH1	151.3 ± 30.3	100.4 ± 33.7	352.8 ± 52.1	116.0 ± 34.1
MPH2	128.3 ± 61.6	93.2 ± 14.9	366.1 ± 60.1	114.0 ± 12.0
MiH1	15.4 ± 10.2	96.8 ± 4.5	170.3 ± 103.4	99.3 ± 6.5
MiH2	0.00	69.5 ± 6.5	134.6 ± 79.8	60.9 ± 7.2
MiH3	0.00	74.8 ± 12.9	0.00	80.1 ± 22.0
MF1	0.00	51.1 ± 27.4	102.0 ± 76.0	30.8 ± 12.2

Table 5: Homogeneous subsets for the subsurface damage at 12 and 40 Kg.

Subset	Subsurface damage at 12 kg					ANOVA F = 15.69, p<0.001			
1	MiH2	MiH3	MF1	MiH1	LP1				
2				MiH1	LP1	MPH2			
3					LP1	MPH2	LP3	MPH1	
4						MPH2	LP3	MPH1	LP2
Subset	Subsurface damage at 40 kg					ANOVA F = 25.96, p<0.001			
1	MiH3	MiH2	MiH1	MF1	MPH1				
2		MiH2	MiH1	MF1	MPH1	MPH2			
3				MF1	MPH1	MPH2	LP1		
4					MPH1	MPH2	LP1	LP3	
5								LP3	LP2

Subsets determined by Scheffe Multiple Comparison Test (Level p= 0.05)

Table 6. Pearson's Correlation Coefficients.

	SURFACE DEFORMATION 12Kg	SURFACE DEFORMATION 40Kg	SUBSURFACE DAMAGE 12Kg	SUBSURFACE DAMAGE 40 Kg
SURFACE HARDNESS	-0.51 $p < 0.001$	-0.34 $p = 0.04$		
SUBSURFACE HARD			0.64 $p < 0.001$	0.73 $p < 0.001$

Table 7 Kendall's tau-c values based on filler size category.

	SURFACE DEFORMATION 12Kg	SURFACE DEFORMATION 40Kg	SUBSURFACE DAMAGE 12Kg	SUBSURFACE DAMAGE 40 Kg	SURFACE HARDNESS	SUBSURFACE HARDNESS
CAT 1-3	0.39 p<0.001	-0.06 p=0.684	0.09 p<0.001	0.84 p<0.001	0.58 p<0.001	0.853 p<0.001

Legend

Figure 1: Light micrograph of resin composites (x200). Incident light illumination with crossed polars.

LP = Large Particle

MPH = Medium Particle Hybrid

MiH = Micro Particle Hybrid

MF = Microfilled

Figure 2: The specimen rig for cyclic compression under water.

Figure 3A Surface deformation and position of microhardness indentations

3B Location of subsurface stain and position of microhardness indentations.

Figure 4 Examples of subsurface damage stained with AgNO_3 after 2000 cycles between 0-12 kg and 0-40 kg.

Figure 5 Scanning Electron Micrograph of Medium Particle Hybrid (MPH1) after 2000 compression cycles between 0 – 40kg. Arrow shows debonding between resin and filler phase.