# Shear bond strength of Resilon to a methacrylate-based root canal sealer

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## Abstract

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**Aim** To evaluate the adhesive strength of Resilon to Next$^{TM}$ root canal sealant (Heraeus–Kulzer), a methacrylate-based root canal sealer, using a modified microshear bond testing design.

**Methodology** Flat Resilon surfaces of different roughnesses (smooth surface and surface roughness equivalent to 320-grit and 180-grit) were prepared by compression moulding for bonding to the sealer and compared with a composite control. The shear strength data were statistically analysed using Kruskal–Wallis one-way ANOVA on ranks and Dunn's multiple comparison tests ($\alpha = 0.05$). After shear testing, fractured specimens were examined using a field emission-scanning electron microscope for detailed analysis of the failure modes.

**Results** The composite control exhibited significantly higher mean shear strength (7.62 MPa) that was 4.4–4.7 times those of the Resilon groups (1.64–1.74 MPa; $P < 0.001$). Increasing the surface roughness of the Resilon surface did not contribute to further improvement in shear bond strength for this methacrylate-based sealer ($P > 0.05$). Failure modes in the composite control were cohesive and mixed failures, while those in the Resilon groups were predominantly adhesive failures, with a small percentage of mixed failures. Ultrastructural evidence of phase separation of polymeric components could be identified in Resilon. Both intact, non deformed and plastically deformed Resilon surfaces could be observed in specimens that exhibited adhesive failures.

**Conclusion** The low shear strength of Resilon to a methacrylate-based sealer compared with a composite control suggests that the amount of dimethacrylate incorporated in this filled, polycaprolactone-based thermoplastic composite may not yet be optimized for effective chemical coupling to methacrylate resins.

**Keywords:** field emission-scanning electron microscope, methacrylate sealer, polycaprolactone, Resilon, shear bond strength.

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## Introduction

Improvements in apical and coronal seals (Saunders & Saunders 1994, Ray & Trope 1995, De Moor & Hommez 2000, Çobankar *et al.* 2004), and strengthening of endodontically treated teeth (Teixeira *et al.* 2004a) have been proposed by establishing monoblocks (i.e. continuum between the root fillings and dentine) via bonding of the root filling materials to intraradicular dentine (Teixeira *et al.* 2004b). This is similar to contemporary adhesive strategies used for intracoronal restorations that attempt to eliminate microleakage and strengthen coronal tooth structures by creating similar monoblocks between tooth substrates and restorative materials. Whereas reasonable

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adhesion to intraradicular dentine may be achieved using etch-and-rinse or self-etch dentine adhesives and compatible methacrylate-based resin cements (Gogos *et al.* 2003, Hayashi *et al.* 2005, Schwartz & Fransman 2005), the creation of endodontic monoblocks has been hampered by the general lack of chemical union between the polyisoprene component of conventional dental gutta-percha and zinc oxide-eugenol, epoxy resin, calcium hydroxide or glass–ionomer-based sealers (Lee *et al.* 2002, Tagger *et al.* 2002, Saleh *et al.* 2003). The recent introduction of Resilon (Resilon Research LLC, Madison, CT, USA) as an alternative root filling material offers the promise of adhesion to root dentine (Shipper *et al.* 2004, Teixeira *et al.* 2004a, Shipper *et al.* 2005). As this filled polycaprolactone polymer contains a blend of dimethacrylates, the manufacturers claim it bonds well to methacrylate-based resin sealers (Jia & Alpert 2003, Jia *et al.* 2005).

Bonding of non resinous restorative materials such as bonded amalgams and silanized ceramics to methacrylate-based resin cements has traditionally been evaluated by comparing the results with those achieved between these cements and resin composites, using the same strength evaluation equipment and testing parameters (Olmez & Ulusu 1995, Shimoe *et al.* 2004). Thus, a realistic test of the strength of the Resilon-sealer bond would be to measure its strength when the sealer is bonded to a standard resin composite control. This comparison is necessary in light of the gaps seen in root canal fillings made with Epiphany/Resilon (Tay *et al.* 2005a). It is thought that these gaps were the result of the inability of the Resilon-sealer bond to resist shrinkage stresses generated during polymerization of the root canal sealer (Alster *et al.* 1997). Thus, the objective of this study was to evaluate the contribution of chemical coupling and micromechanical retention to the adhesive strength of Resilon to a methacrylate-based sealer. The hypothesis tested was that the shear strength of a methacrylate-based root canal sealer to Resilon is similar to that of the sealer to a resin composite.

## Materials and methods

### Preparation of Resilon and resin composite disks

Resilon pellets (Pentron Clinical Technologies, Wallingford, CT, USA) were purchased from the manufacturer. They were heat moulded into 0.5 mm thick circular disks of 7 mm in diameter to provide flat bonding surfaces with different surface roughness. Three Resilon groups were created:

*Smooth surface*
Resilon pellets were first plasticized in a laboratory dry-heating oven at 80 °C. The melted pellets were sandwiched between top and bottom Mylar films (DuPont Corp., Wilmington, DE, USA) and two preheated glass slabs. Compression moulding was performed inside the oven by placing a 5 kg weight over the top glass slab (Tay *et al.* 2005b). Resilon sheets 0.5 mm thick were created by inserting 0.5 mm thick Teflon spacers on either side of the plasticized pellets. After cooling to ambient temperature, the Mylar films were peeled off, revealing shiny polymer surfaces. Thirty Resilon disks of 7 mm in diameter were created from these sheets using a metal punch (Small Parts Inc., Miami Lakes, FL, USA) and a mallet.

*Rough surface (320-grit)*
The above protocol was repeated with the top Mylar film replaced by a piece of 320-grit silicon carbide paper (i.e. 32–36 $\mu$m diameter particle roughness).

*Rough surface (180-grit)*
The above protocol was repeated with the top Mylar film being replaced by a piece of 180-grit silicon carbide paper (i.e. 76 $\mu$m diameter particle roughness).

As bond strength is not a material property and is dependent on the testing methods, a control group consisting of resin composite disks was used to obtain shear strength data with which the bonding efficacy of Resilon may be compared. A microhybrid composite (Gradia Direct; GC Corp., Tokyo, Japan) was sandwiched between top and bottom Mylar films and unheated glass slabs. The composite was compressed in 0.5 mm thick Teflon moulds with prepunched 7 mm diameter holes and light-cured for 40 s from the top and subsequently from the bottom, to create circular disks with smooth bonding surfaces that were devoid of oxygen inhibition layers. Subsequent bonding was performed within 2 h to take advantage of existing free radicals within the freshly polymerized, oxygen inhibition layer-free composite (Suh *et al.* 2003).

### Bonding procedures

A modified microshear bond testing protocol was employed for examining the adhesion of Next$^{TM}$

(Heraeus–Kulzer, Hanau, Germany), a methacrylate-based root canal sealant, to Resilon. Accordingly, 5 mm long segments of a translucent polyurethane tubing (Small Parts Inc.) with an internal diameter of 3.25 mm was used in lieu of the 0.7 mm diameter Tygon tubing originally employed by McDonough *et al.* (2002) [Fig. 1(A-a)], because the sealer was too viscous to enter the smaller tubing. Gradia Direct resin composite was inserted into these tubings and light-cured incrementally to produce composite cylinders with smooth and flat bases [Fig. 1(A-b)].

Each composite cylinder was placed on a Resilon or composite disk, with its flat base in contact with the disk surface. A layer of nail varnish was applied to the rest of the disk surface [Fig. 1(A-c)] to create a standardized, circular bonding substrate area and to avoid subsequent smearing of the remaining substrate surface by the root canal sealer that would inadvertently increase the bond strength measurements [Fig. 1(A-d)]. Upon drying of the varnish, the empty polyurethane tube was placed over the exposed bonding surface. Next$^{TM}$ bonding agents A and B were mixed and applied to the base of the composite cylinder to create a strong bond between the base of the composite cylinder and the root canal sealer, directing the failure to occur along the disk-sealer interface.

The Next$^{TM}$ root canal sealant was mixed and dispensed via an auto-mixing tip into the polyurethane tubing [Fig. 1(A-e)]. The adhesive-coated side of the composite cylinder was reinserted with light pressure into the tubing to displace the root canal sealer onto the Resilon surface. A 0.5 mm thick layer of sealer was created by measuring the length of the composite cylinder that extruded out of the polyurethane tubing [Fig. 1(A-f)]. As the tubing was translucent, the dual-cured sealer was photo-irradiated through the tubing from four sides for 60 s each. The assembly was left in this condition for 24 h to ensure optimal polymerization of the resin sealer via additional auto-curing. After removing the polyurethane tubing [Fig. 1(A-f)], each bonded sample was inspected under a stereomicroscope (SMZ-10, Nikon; Tokyo, Japan) at 20× magnification.

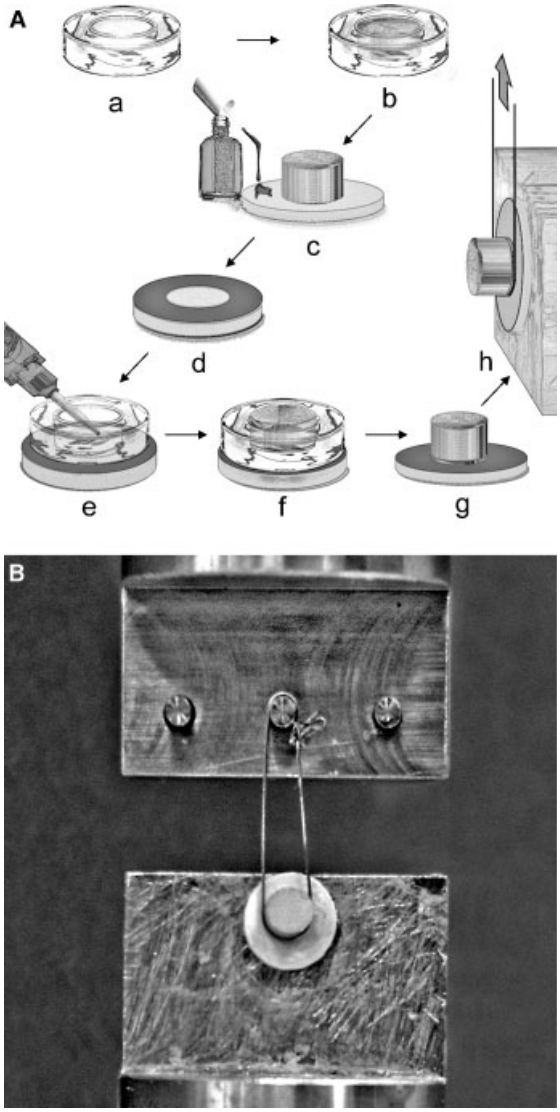


**Figure 1** (A) Schematic representation of the modified micro-shear bond testing protocol employed for evaluation of the adhesion of methacrylate-based root canal sealers to Resilon. (a) Short polyurethane (PE) tubing segment with an internal diameter of 3.25 mm. (b) Preparation of composite cylinder inside the PE tubing. (c) Placement of composite cylinder on Resilon disk (R) and application of nail polish. (d) Space that was left behind for bonding after removal of the composite cylinder from the R. (e) Empty tubing segment placed over exposed Resilon surface and filled with a methacrylate-based root canal sealer. (f) Insertion of the bonding agent-coated composite cylinder into the polyurethane tubing to create a 0.5 mm thick layer of root canal sealer between the cylinder and the Resilon disk. (g) Removal of the flexible polyurethane tubing after 48 h to ensure optimal polymerization of the root canal sealer. (h) Placement of an orthodontic wire as close as possible to the attached composite cylinder and stressing the bonded assembly to failure in a universal testing machine. (B) A photograph illustrating the attachment of an orthodontic wire to the base of the bonded composite cylinder during bond strength testing.

Specimens with bonding defects such as voids, incomplete coverage of the exposed bonding substrate area or visually apparent interfacial gaps were excluded. The best 25 of the 30 bonded specimens in each group were selected by inspection with the microscope to undergo bond testing.

### Microshear tesing

Each bonded disk of Resilon or composite was secured with cyanoacrylate glue (Zapit; DVA, Corona, CA, USA) to a fixture that was screwed into the base and aligned with the loading axis of a Bencor Multi-T testing assembly (Danville Engineering, San Ramon, CA, USA). A wire loop prepared from an orthodontic stainless steel ligature wire (0.41 mm in diameter) was wrapped around the bonded assembly so that it was as close as possible to the base of the resin sealer [Fig. 1(A-h, B)]. A tensile load was applied via a universal testing machine (Model 4440; Instron Inc., Canton, MA, USA) at a crosshead speed of 1 mm $min^{-1}$. The relatively slow crosshead speed was selected in order to produce a shearing force that resulted in debonding of the composite cylinder along the disk-sealer interface. Debonded specimens were initially examined with the stereomicroscope at 20× magnification for determination of the failure mode. Failure was classified as adhesive, mixed, or cohesive within the Resilon or composite disks.

Interfacial shear strength was calculated by dividing the maximum load recorded on failure by the circular bonding area in $mm^2$ and expressed in MPa. Specimens that failed prematurely during handling were assigned null strength values and included in the statistical analysis. As the normally distributed (Kolmogorov–Smirnof test) data exhibited unequal variance (Levene median test), they were statistically analysed with Kruskal–Wallis one-way ANOVA on ranks and Dunn's multiple comparison tests, with statistical significance set at $\alpha = 0.05$.

### Fractographic analysis

Representative debonded composite cylinders and the corresponding Resilon/composite disks from each group were sputter-coated with gold/palladium for examination with a field emission-scanning electron microscope [(FE-SEM); Leo 1530 Gemini; Leo Electron Microscopy Ltd, Zeiss, Oberkochen, Germany]. The specimens were examined with accelerating voltages of 20 keV to identify both surface and subsurface features and 3 keV to identify the topographical features without interference from the subsurface filler particles that were present within the disk specimens (Tay *et al.* 2005b). Images were taken with either the secondary electron mode or in-lens mode of the microscope.

## Results

The composite control exhibited significantly higher mean shear bond strength that was 4.4–4.7 times those of the Resilon groups ($P < 0.001$; Table 1). Increasing the surface roughness of the Resilon surface did not contribute to further improvement in shear bond strength for this methacrylate-based sealer ($P > 0.05$). Failure modes in the composite control were predominantly cohesive and mixed failures, while those in the Resilon groups were predominantly adhesive failures, with a small percentage of mixed failures (Table 1).

Field emission-scanning electron microscope examination confirmed the existence of mixed [Fig. 2(A)] and cohesive failure modes that were characteristic of the composite control group. Exposed surfaces of the fractured root canal sealer revealed the presence of larger particulate and smaller fumed silica fillers [Fig. 2(B)]. In mixed failures of the Resilon smooth group [Fig. 3(A)], plate-shaped fillers were identified with the intact Resilon material [Fig. 3(B)]. In addition, globular domains could be observed within the Resilon matrix that could be better visualized when the specimens were examined with the in-lens mode at

**Table 1** Shear bond strengths of Next™ Root Canal Sealant (Heraeus–Kulzer) to resin composite (control) and Resilon

| Groups ($n = 25$) | Shear bond strength (MPa)[a] | Failure mode (stereomicroscopy) | | | Number of premature failure[b] |
|---|---|---|---|---|---|
| | | Cohesive | Mixed | Adhesive | |
| Resin composite smooth surface (control) | 7.62 [1.42]$^{A}$ | 8 | 16 | 1 | 0 |
| Resilon smooth surface | 1.64 [0.67]$^{B}$ | 0 | 5 | 20 | 1 |
| Resilon rough surface (320-grit) | 1.74 [0.67]$^{B}$ | 0 | 2 | 23 | 0 |
| Resilon rough surface (180-grit) | 1.67 [0.63]$^{B}$ | 0 | 3 | 22 | 0 |

[a]Values are means (SD). Groups with the same upper case letters within the column are not statistically significant ($P > 0.05$).
[b]Premature failures were assigned null bond strength values and were included in the statistical analysis.

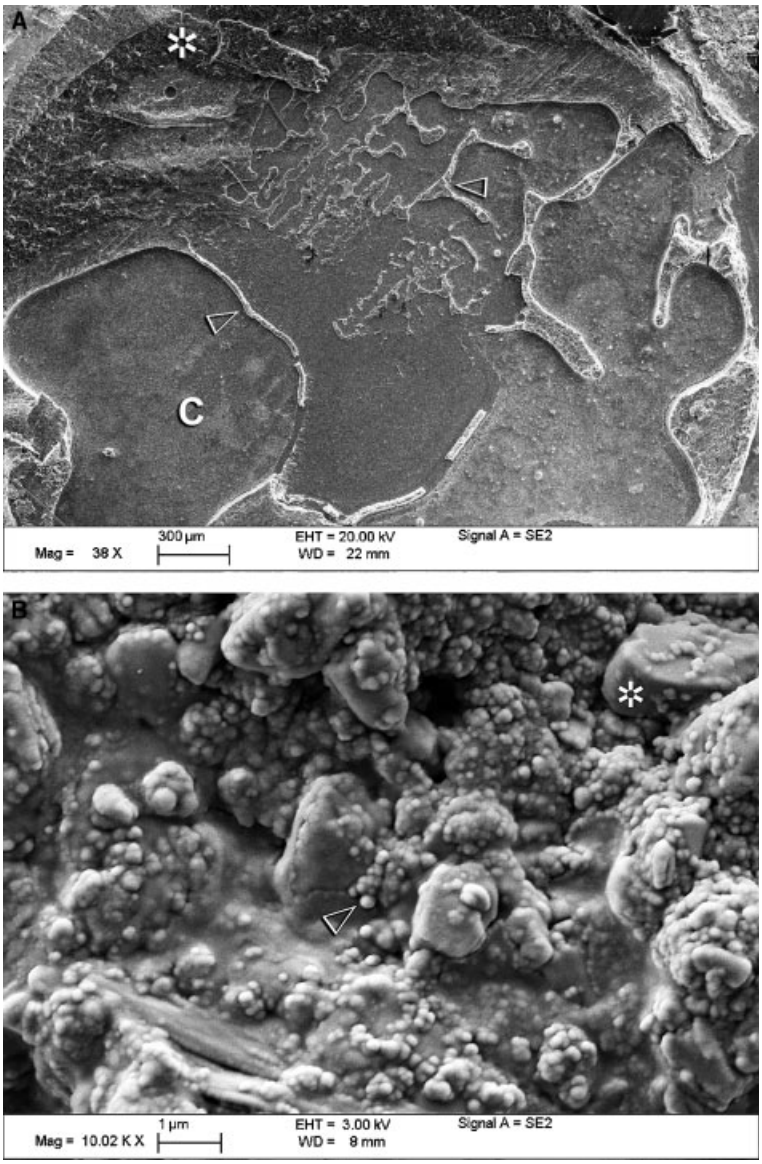


**Figure 2** FE-SEM micrographs of the composite smooth group. (A) A low magnification view, taken at 20 keV, of a mixed failure mode that involved failure within the composite cylinder (asterisk) and the root canal sealer (open arrowheads). C, composite disk. (B) A high magnification view, taken at 3 keV, of the particulate fillers (*) and fumed silica (open arrowhead) that were present within the fractured resin sealer.

low keV to avoid the interference from the subsurface fillers [Fig. 3(C)].

Specimens in the Resilon smooth group that were classified as adhesive failures by stereomicroscopical microscopy were found to contain structurally deformed areas on the Resilon surface when they were examined using FE-SEM [Fig. 4(A)]. At higher magnifications, these areas represented plastically deformed Resilon matrix in which the lamellae of the semicrystalline polycaprolactone component became highly aligned after stretching [Fig. 4(B)]. These plastically deformed regions were filler-sparse when compared with the underlying non deformed regions [Fig. 4(C)].

Specimens from the Resilon-320 grit and Resilon-180 grit groups contained surface holes created by compression moulding with silicon carbide papers [Fig. 5(A, D)]. Similar plastic deformation occurred along the periphery of these surface asperities [Fig. 5(B)], resulting in filler-sparse Resilon matrices [Fig. 5(C)].

## Discussion

Different types of methacrylate-based sealers are commercially available for the coupling of Resilon to root dentine. They include Epiphany (Pentron Clinical

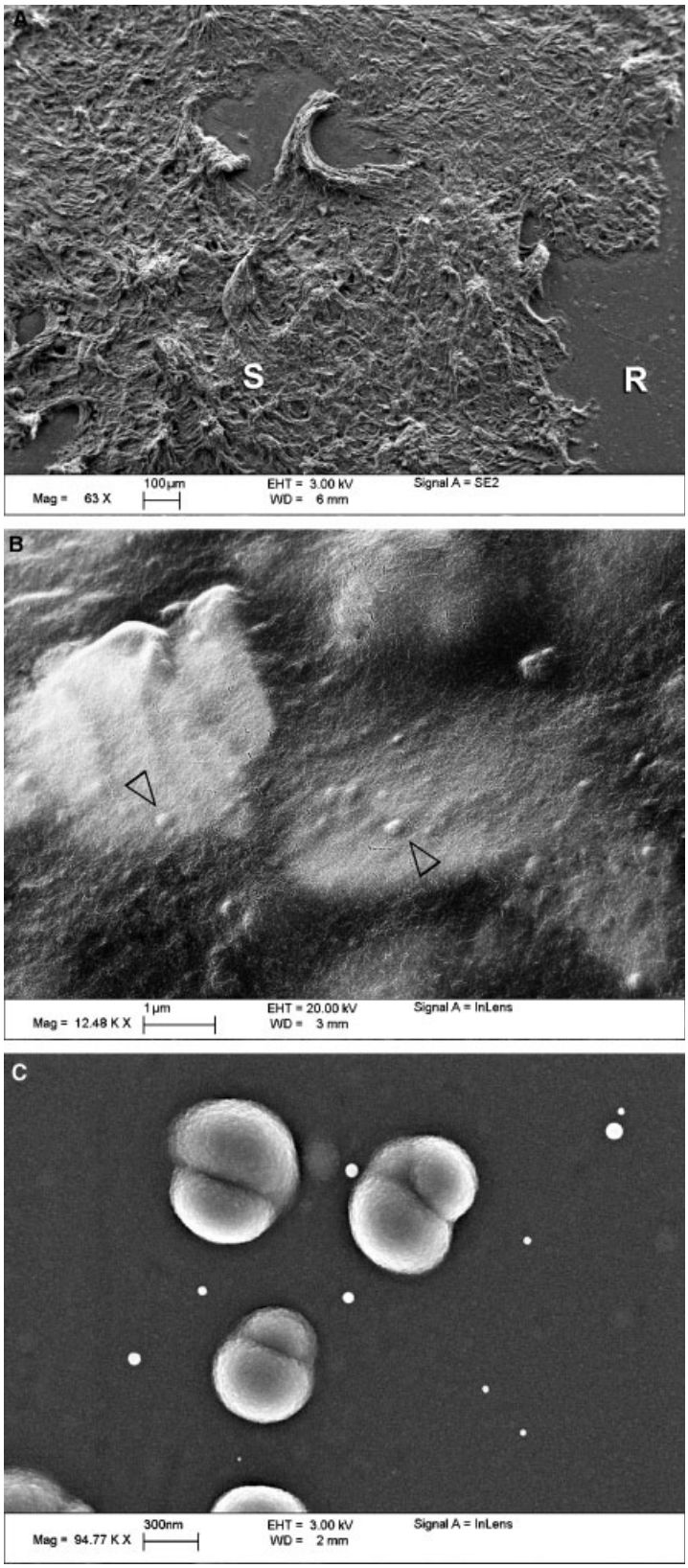


**Figure 3** Field emission-scanning electron microscope micrographs of specimens that were classified as mixed failure by stereomicroscopical examination in the Resilon smooth group. (A) A low magnification view (3 keV) of a fractured specimen surface showing the fractured root canal sealer (S) and an intact Resilon surface (R). (B) A high magnification view (20 keV) of the smooth Resilon surface showing the presence of plate-shaped subsurface fillers beneath the surface polymer matrix. Globular domains (open arrowheads), probably representing phase separation of the dimethacrylate component in the polycaprolactone-based Resilon material, could be identified within the polymer matrix. (C) A very high magnification view (3 keV) of the surface of the polymer matrix. Without the interference from the subsurface fillers, the globular domains could be more clearly visualized.

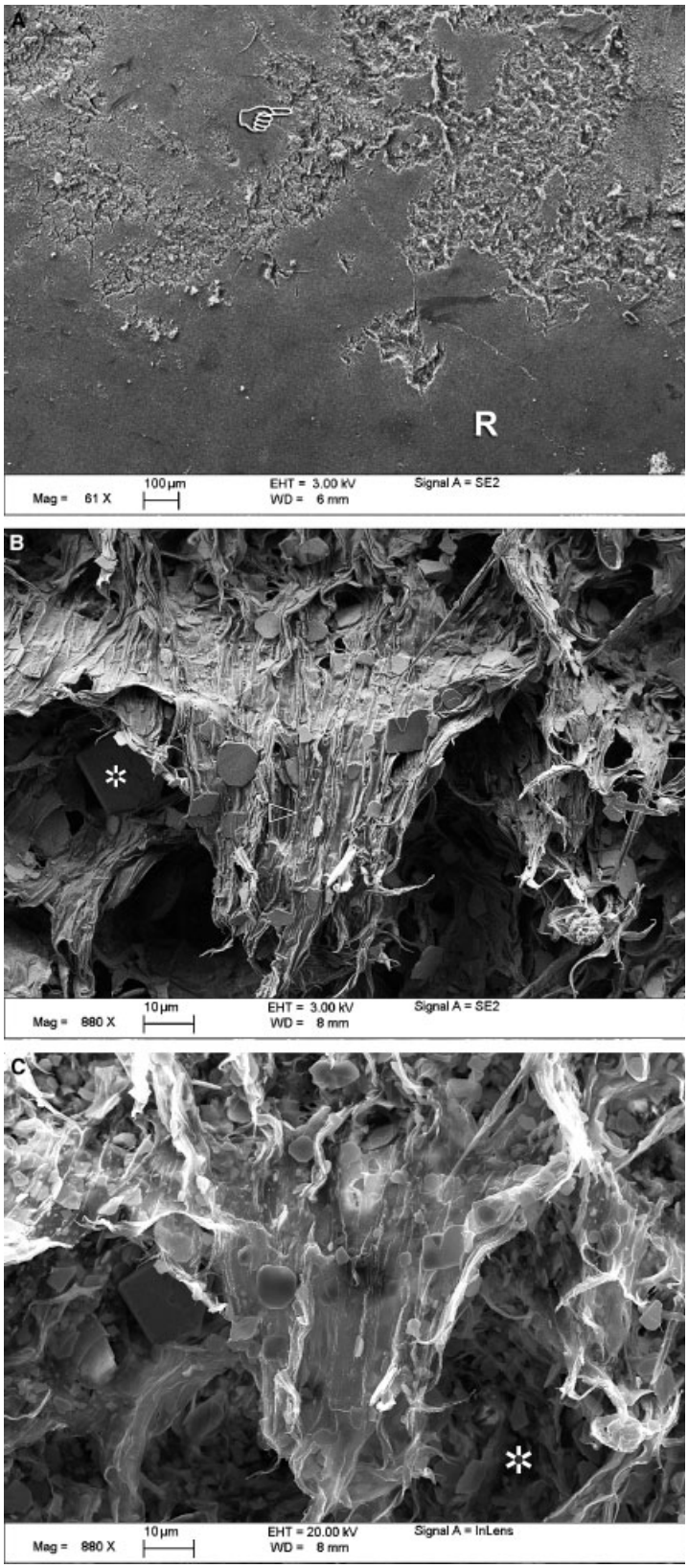


**Figure 4** Field emission-scanning electron microscope micrographs of specimens that were classified as adhesive failure by stereomicroscopical examination in the Resilon smooth group. (A) A low magnification view (3 keV) of a fractured specimen surface showing a predominantly smooth Resilon surface (R) that contained patches (pointer) wherein structural deformation had occurred after debonding. (B). A high magnification view (3 keV) of the structurally deformed Resilon surface, depicting only the surface features without interference from the subsurface fillers. Loose, plate-shaped fillers (*) could be readily identified. In addition, plastic deformation of the Resilon polymer matrix resulted in an almost parallel alignment (open arrowhead) of the lamellae of the semi-crystalline polycaprolactone component of the Resilon polymer matrix. (C) The same high magnification view, taken at 20 keV, showing the presence of very few plate-shaped fillers within the plastically-deformed portion of the Resilon matrix. By contrast, the non deformed, underlying Resilon material (*) revealed a much higher density of the subsurface plate-shaped fillers.

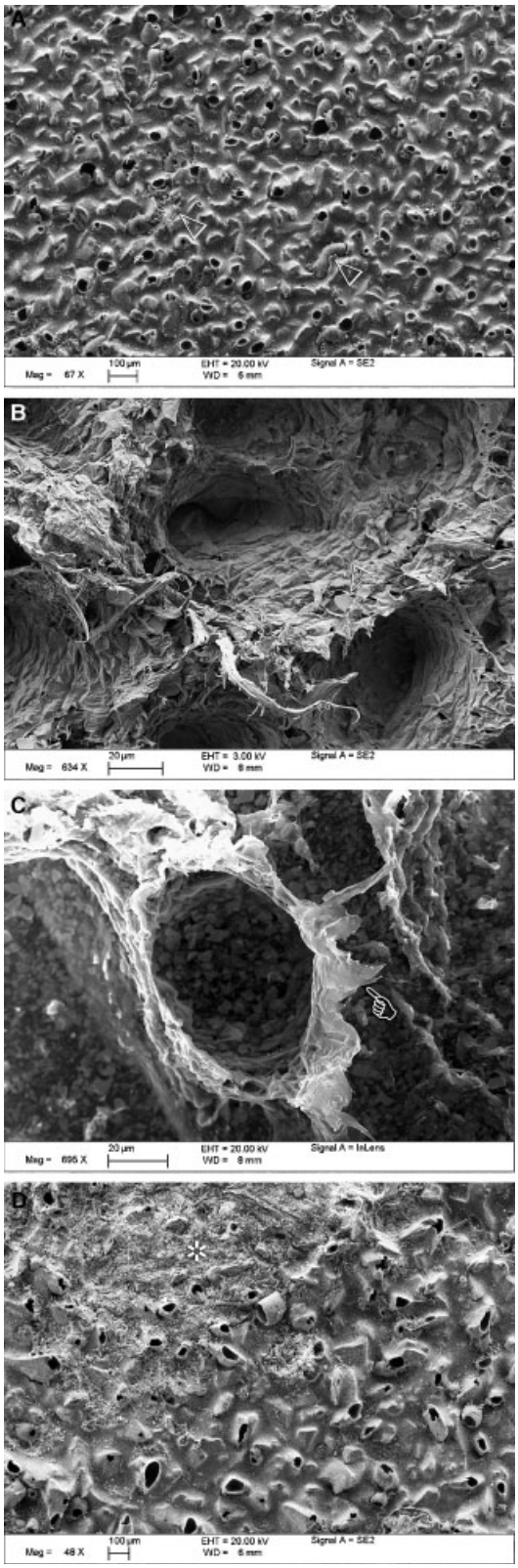


**Figure 5** Field emission-scanning electron microscope micrographs of representative debonded specimens from the Resilon 320-grit and 180-grit groups. (A) A low magnification view (20 keV) of a specimen from the Resilon 320-grit group showing the creation of 20–50 µm wide holes on the Resilon disk surface. The specimen was classified as an adhesive failure on stereoscopical examination. Very little remnant resin sealer (open arrowheads) was trapped within the surface asperities. (B) A high magnification view (3 keV) showing the surface characteristics of the debonded Resilon surface where remnant resin sealer was absent. Loose plate-shaped fillers (open arrowhead) could be identified along the plastically-deformed periphery of the holes. (C) A high magnification view (20 keV) comparing the filler-sparse, plastically-deformed Resilon matrix along the periphery of these holes (pointer), and the filler-dense, non deformed Resilon material. (D) A low magnification view (20 keV) of a specimen from the Resilon 180-grit group that was classified as a mixed failure on stereoscopical examination. 50–100 µm wide holes were created on the Resilon disk surface. Large patches of fractured resin sealer (*) were identified along the Resilon surface. Similar plastic deformation around the periphery of the surface asperities could be observed at high magnifications.

Technologies, Wallingford, CT, USA), RealSeal (Sybron Kerr, Orange, CA, USA), SimpliFill (LightSpeed, San Antonio, TX, USA) and Next$^{TM}$. The Next$^{TM}$ obturation system differs from the other three in that it uses a Resilon-capped fibreglass obturator (Tapered Obturator and Post-Obturator, Heraeus–Kulzer) for immediate core build-up after obturation of the root canals that is analogous to Pentron's FiberFill system (Shipper & Trope 2004). In this study, only the bondability of the apical Resilon portion of the Next$^{TM}$ obturation system to the proprietary root canal sealer was examined, so that additional results eventually obtained for the coupling of Resilon to other three Resilon-associated methacrylate-based sealers may be compared.

The modified microshear bond testing design was employed as all Resilon specimens prepared with conventional microtensile (Erdemir *et al.* 2004) or microshear techniques (Giannini *et al.* 2004) exhibited premature failures in previously conducted pilot studies. As the shear bond strength of the Next$^{TM}$ root canal sealant is significantly lower than the resin composite control, this led to the conclusion that chemical coupling of the methacrylate-based sealer to Resilon is weak despite the observation of plastic deformation of the Resilon matrix. For this particular root canal sealer, increasing the surface roughness of the Resilon material did not result in improvements in shear bond strength. This is in contrast with the results obtained

for RealSeal (Sybron Endo, Orange, CA, USA), another Resilon-compatible methacrylate-based root canal sealer under the same experimental testing conditions (Tay FR, Hiraishi N, Pashley DH, Loushine RS, Weller RN, Gillespie WT, Doyle MD, unpublished results). Indeed, the mean shear bond strengths of Next$^{TM}$ to the composite control and the Resilon smooth groups were 1.9 and 10.8 times respectively of the corresponding shear bond strengths of RealSeal to these groups. Thus, the improved chemical coupling of Next$^{TM}$ to Resilon smooth surfaces could have compensated for the additional contribution of mechanical retention via the creation of surface asperities in the Resilon 320- and 180-grit groups.

The observation of highly oriented lamellae arrangement within the semi-crystalline (Harrison & Jenkins 2004) polycaprolactone component of Resilon after it was stretched to failure represents a feature that is commonly observed in the deformation of elastomeric matrices that contain spherulitic structures. Rearrangement of the crystalline and amorphous regions of the spherulites occurs when these polymers are subjected to stresses, such as those applied during cold drawing of the polymer (Ward & Hadley 1997). These changes are apparent to the naked eye as necking of the plastically deformed regions. In these regions craze lines are present that are perpendicular to the applied stresses (Ward & Hadley 1997). Ultrastructurally, re-orientation of the lamellae regions with less highly ordered polymer chains occur at low stresses. This is followed by the almost parallel arrangement of the lamellae regions upon the application of higher stresses that result in the physical appearance of necking and crazing, until the material yields with ductile failure (McLean & Sauer 1999, Michler & Godehardt 2000).

Phase separation of components is common in polymer blends prepared with mutually immiscible monomers (Na *et al.* 2002, Wang & Composto 2003, Mano *et al.* 2004). Considering that polycaprolactone is the major and urethane dimethacrylate the minor polymeric component in Resilon (Jia & Alpert 2003, Jia *et al.* 2005), probably in a ratio of approximately 10 : 1 (Jia 2005), the phase separation in the form of globular domains within the Resilon matrix may represent an emulsified dimethacrylate phase within a continuous polycaprolactone phase. Although chemical coupling of Next$^{TM}$ to Resilon was evident by the appearance of plastic deformation of the Resilon matrix, there were areas in which smooth intact Resilon surfaces remained after debonding. Thus, it appears that the amount or method of dimethacrylate incorporated in Resilon may not yet be optimized for effective and predictable chemical coupling to methacrylate-based sealers.

As the apical Resilon portion of the Next$^{TM}$ obturator is not amendable to light-curing, unlike the coronal fibreglass portion, the use of slow auto-curing dynamics in the dual-cured root canal sealer may be considered an advantage in minimizing shrinkage stress build-up that favours the survival of the Resilon-sealer bonds. However, in view of the extremely high C-factors encountered in long, narrow root canals (Goracci *et al.* 2004, Tay *et al.* 2005c), it is dubious whether the very weak Resilon-sealer bonds are capable of resisting polymerization shrinkage stresses that develop during the setting of the resin sealer. This issue becomes even more pressing when the dual-cured sealer is light-cured from a root-filled canal orifice to create an immediate coronal seal of the fibreglass obturator with root dentine, because this prevents stress relief by resin flow (Ferracane 2005).

The latest pending patent on the Resilon root filling material described an experimental version of this material that utilized low fusion polycaprolactones and urethane dimethacrylate as an inner polymeric core and high fusion polycaprolactones and urethane dimethacrylate as an outer polymeric sheath. Bioactive glass, barium sulphate, bismuth oxychloride and 'red iron oxide' were incorporated as fillers in both the inner core and outer sheath (Jia 2005). The rationale of using an integrated core and sheath with differential 'melt flow indices' was to provide an inner core with similar strength and rigidity as the commercial Resilon version and an outer sheath with increased mouldability and forming capability (Jia 2005). Such a modification implies that the core-sheath version has to be used with cold lateral compaction techniques or as an integral part of a root filling/fibreglass post-obturator system. However, incorporating a dimethacrylate in polycaprolactone is not the only means by which chemical coupling may be achieved between root filling materials and sealers. An alternative strategy that integrates a core-sheath design involves the use of gutta-percha cones that are coated with a polybutadiene-diisocyanate-methacrylate resin (Haschke 2004). This strategy has merits in that comparatively inert gutta-percha is employed, in lieu of the bacterial enzyme-degradable polycaprolactone component that is utilized in Resilon (Tay *et al.* 2005d). The bond strength of resin-coated gutta-percha (Ultradent, South Jordan, UT, USA) to methacrylate-based root canal sealers is currently being investigated using the modified microshear bond testing protocol developed in this work.

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