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Gaps at the interface between dentine and self-adhesive resin cement in post-endodontic restorations quantified in 3D by phase contrast-enhanced micro-CT

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Abstract

Soares AP, Bitter K, Lagrange A, Rack A, Shemesh H, Zaslansky P. Gaps at the interface between dentine and self-adhesive resin cement in post-endodontic restorations quantified in 3D by phase contrast-enhanced micro-CT. *International Endodontic Journal*, **53**, 392–402, 2020.

Aim To assess the extent of gaps between root dentine and titanium or fibreglass post restorations following cementation with a self-adhesive resin cement.

Methodology Fourteen root filled maxillary central incisors restored with prefabricated posts made of Fibreglass (n = 7) or Titanium (n = 7) and cemented with RelyX Unicem 2 were imaged by rapid, high-resolution phase contrast-enhanced micro-CT (PCE-CT) in a synchrotron X-ray imaging facility (ID19, ESRF, 34 KeV, 0.65 μm pixel resolution). Reconstructions were used to measure canal, cement and post perimeters and cross-sectional areas and interfacial gaps at 0.1 mm increments in the root canal space, along the cervical region of the tooth. Remnants of endodontic sealer (AH Plus), when present, were also quantified. Mann–Whitney and 2-way ANOVA tests were used to compare findings within slices and between the two post groups. Pearson correlation coefficients (r) were determined between the interfacial gaps and the other measured parameters.

Results Clearly detectable gaps were found in 45% (±14%) of the interfaces between dentine and cement, along the canal in the cervical area of the tooth beneath the core. The length of interfacial gaps was moderately correlated to the canal cross-sectional area, to the canal perimeter and to the canal area filled by cement ($R = 0.52 \sim 0.55$, $P < 0.001$). There was no significant difference between samples with fibreglass or titanium ($P > 0.01$). Both post types had defect-free interfaces with cement. Endodontic sealer remnants were found on ~10% of the canal walls and were moderately correlated to the presence of gaps. Approximately 30% of the sealer-affected interfaces exhibited no detachment between dentine, sealer and cement.

Conclusions Self-adhesive cements had interfacial gaps along substantial regions of the root canal surface, which was not correlated with the amount of cement in the canal. PCE-CT proved to be an excellent non-destructive method to study root canal restorations of hydrated samples in 3D.

Keywords: cement debonding, phase contrast enhanced microcomputer tomography, post and core restoration, self-adhesive composite cement.

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Introduction

Self-adhesive resin cements are used routinely to cement posts in root filled teeth. Their main advantage over other resin cements is that they adhere directly to the tooth structure without additional treatment steps of etching, conditioning or adhesive application (Burke *et al.* 2006). Indeed, standardized auto-mixing cement kits and thin intraoral tips have improved intracanal cement delivery (Opdam *et al.* 2002). Previous experiments found that the performance of self-adhesive resins is at least as good as other types of dental cements for post luting, using various methods of analysis including push-out bonding strength tests (Zicari *et al.* 2008, Pereira *et al.* 2014, Bitter *et al.* 2017). Despite their many advantages, self-adhesive cements have several limitations, such as occasional loss of retention and leakage that might lead to failure of the root canal treatment (Rasimick *et al.* 2010). Whilst some researchers (Kirkevang *et al.* 2000, Tavares *et al.* 2009) maintain that the apical seal is as important as the coronal seal for the longevity of the restoration, others claim that a well-sealed crown is more important (Ray & Trope 1995). Either way, it is accepted that a permeable restoration situated coronal to the root canal filling may lead to penetration of fluid and bacteria via microleakage (Neves *et al.* 2014). Due to the causative link between canal infections and periapical lesions (Kakehashi *et al.* 1966), interfacial gaps and microleakage in root canal restorations are undesirable and should be avoided (Heintze 2013, Jokstad 2016). Laboratory research has found that post cementation with self-adhesive resins has a larger number of voids in the cervical third of the root canal as compared with the middle or apical thirds (Uzun *et al.* 2016, Pedreira *et al.* 2016). All these observations raise concerns regarding the capacity of self-adhesive cements to create a defect-free, tight seal of the coronal third of the root canal.

Whilst it is desirable to attain a homogeneous seal between the restoration and the canal walls, there are no standardized ways to investigate and quantify the integrity of this resin–dentine interface. Push-out or pull-out mechanical tests are frequently used to assess the durability of post bond to canal dentine (Heintze 2013). However, the relationship between bond strength and structural integrity of the interface between dentine and cement is still uncertain (Rasimick *et al.* 2010) even though it is assumed that reduced strength values are suggestive of the presence of interfacial gaps at the canal walls.

Different approaches exist to directly examine the integrity of the interface between root filling components. Traditionally, interfaces between tooth tissues and fillings were visualized by serial sectioning and microscopy observations, for example stereo-, scanning electron- or confocal laser scanning-microscopes (Heintze 2013). Although informative, all these methods are destructive, as they require physical sectioning of the sample, which may inadvertently cause cracks to appear (Zaslansky *et al.* 2011). Indeed, the number of voids or the extent of interfacial spaces might be underestimated due to smear layer or sample preparation damage. An important approach to investigate interfacial gaps uses dyes to study microleakage (Neves *et al.* 2014). It is, however, controversial because of the questionable repeatability of this method. Tracer dyes such as organic colourants or silver nitrate require dye permeation through the filling (De Munck *et al.* 2005), which typically implies that 'through-and-through' gaps must exist (Hilton 2002). Such studies are frequently qualitative, typically focusing on one slice with little or no depth (three dimensional – 3D) information (Neves *et al.* 2014, Carrera *et al.* 2015). Overall, tracer infiltration is never uniform, and it frequently includes 'false-positive' findings as the dye either enters normal openings (e.g. dentine tubules or lateral canals) or reacts with the filling material leading to an overestimation of the presence of gaps (Shemesh *et al.* 2008, Kriznar *et al.* 2018).

Common to the methods listed above is that they are essentially destructive requiring cross-sections to expose 2D surfaces in the 3D volume of the tooth. However, for studying restoration interfaces, nondestructive 3D approaches that maintain and reveal the spatial integrity are of great value. Volume imaging techniques based on tomography have been used in interfacial gap studies (Bakhsh *et al.* 2011, Kwon & Park 2012, Carrera *et al.* 2015). Yet, visibility of details is a serious concern. Due to limited resolution, it is often difficult to distinguish between materials placed deep inside the dense structures of restored roots. The low density of most polymers precludes visualization of internal interfaces, to the extent that some researchers still use silver nitrate as a radiopaque tracer, to assess marginal gaps with laboratory micro-computed tomography (μCT) (Kwon & Park 2012, Neves *et al.* 2014). A different method, swept source optical coherence tomography, uses laser light rather than X-rays such that interfacial defects are easier to see (Nakagawa *et al.* 2013). Optical coherence tomography is, however, limited by strong absorption by tooth/restoration tissues such that regions of interest must be situated near the detector (Nakagawa *et al.* 2013). Overall, the optical signal is rather weak, and the results become difficult to standardize (Bakhsh *et al.* 2011).

Phase contrast-enhanced μCT (PCE-CT) has matured to become a valuable 3D measurement method, well suited to provide accurate 3D information about the presence and extent of voids deep within root canal fillings (Zaslansky *et al.* 2011, Moinzadeh *et al.* 2016). Interfaces observed by PCE-CT are accentuated due to the use of partial coherence X-rays, leading to easier differentiation between material interfaces in root canal fillings. Thus, PCE-CT is useful to image, in 3D, interfacial gaps in a nondestructive manner and at micrometer resolution without requiring tracer penetration. PCE-CT requires the use of specialized high-flux X-ray facilities, able to produce 'laser-like' high-energy X-ray beams, known as synchrotrons (Moinzadeh *et al.* 2016). Such imaging allows fast scanning, that can be tuned to circumvent heating or dehydration of the samples. Yet synchrotron imaging entails overcoming the difficulties of accessing these research facilities, the restricted time allocated per experiment, and the large amount of data needing processing.

In the present *ex vivo* study, the aim was to nondestructively examine interfaces at the micrometer length scale, within intact post and core restorations placed in root filled teeth, made with a widely used self-adhesive resin cement (RelyX Unicem 2, 3M ESPE).

Materials and methods

Samples selection and endodontic preparation

Human maxillary central incisors ($n = 14$) were obtained with written informed consent and requirements of anonymity, under an ethics-approved protocol (EA4/102/14) by the Ethical Review Committee of the Charité Universitätsmedizin Berlin, Germany and stored in 0.5% chloramine T solution. The teeth selected contained no cavities, restorations or fractures and all had a single root 15 ± 1 mm long and a cervical diameter of 7 ± 0.5 mm. An impression of each crown (Silaplast Futur, Detax, Ettlingen, Germany) was obtained for later reconstruction of crown morphology, and all teeth were decoronated at the cervical margin using a water-cooled diamond band saw (Exact 312, Exakt, Norderstedt, Germany). During all treatment steps, roots were continuously kept wet using conventional dental chair waterlines. Each root canal was instrumented as follows: canal orifices were widened using Gates Glidden burs (no. 1-3, VDW, Munich, Germany). Working length was established with a size 10 K-file up to 1 mm from the anatomical apex. Canal instrumentation was carried out with the ProTaper Next System (Dentsply Sirona, Ballaigues, Switzerland) using files X1-X4 up to a final preparation of size 40, 0.07 taper. After each file, an intracanal irrigation of 1% sodium hypochlorite solution (Hedinger GmbH, Stuttgart, Germany) was applied using a Luer Syringe (Dentsply Sirona) and irrigating needles 30 gauge (Max-I-Probe, Dentsply Sirona). For final irrigation, a saline solution was used (Braun, Melsungen, Germany), followed by drying with paper points (ProTaper Next System X4 Paper Points, Dentsply Sirona). Each canal was filled with a single cone of matching gutta-percha (ProTaper Next System X4 gutta-percha cone, Dentsply Sirona) fitted to the working length and evenly coated with endodontic sealer (AH Plus, mixing ratio 1:1, Dentsply Sirona) uniformly spread on the canal walls up to ~7 mm from the apex. The filling was vertically condensed down to 7 mm below the cervical margin using the Calamus Dual System (Dentsply Sirona). Each root was temporarily sealed with Teflon tape covered by a thin layer of temporary filling and stored moist in a water-tight vial for final restoration within 24 h.

Post build-up and restoration

Following storage, gutta-percha and dentine were removed from the canal using standardized pilot and reamer drills (ER System Pilot bur and system reamer, stainless steel, size 090 Komet, Lemgo, Germany) under constant water irrigation to a depth of 9 mm. In this manner, it was possible to ensure that at least 5 mm of apical seal of the root filling remained intact whilst creating standardized post spaces in the coronal region. Each canal was examined under a stereomicroscope (WilD M3Z, Wild Heerbrug, Heerbrug, Switzerland) to identify and remove any visible remaining gutta-percha and all signs of endodontic sealer. Thereafter, 1% sodium hypochlorite was used and flushed with distilled water, followed by blot-drying with paper cones, as recommended by the cement manufacturer. The teeth, divided into two groups, were immediately treated: 7 teeth in group 1 were restored with a fibreglass post (Dentin Post size 090, Komet, Lemgo, Germany) as follows: each post was cleaned with 96% ethanol, silanized (Ceramic Bond, Voco, Cuxhafen, Germany) coated with and cemented using the dual-curing self-adhesive resin cement RelyX Unicem 2 (Automix delivery system, 3M ESPE Dental Products, St Paul, MN, USA). Cement was applied into the root canal using mixing tips coupled with the dedicated intraoral application kit tips. The second group of 7 teeth was similarly restored, but using titanium posts (Kopfstift Titanium Post size 090, Komet). For both groups, chemical polymerization was enhanced by light curing, illuminating the filled canal orifice with a light cure LED (Valo Corded, Ultradent Products Inc, South Jordan, UT, USA) for 40 s. Following polymerization, excess cement was removed with a sharp blade. Each tooth crown was immediately restored with composite (Rebilda DC Quickmix, Voco, Cuxhaven, Germany): the exposed tooth cervical area was etched with phosphoric acid 37% (Orbi Flow, Orbis Dental, Münster, Germany), washed and briefly air-dried. An adhesive system (Futurabond U Single Dose, Voco) was applied for 20 s, agitated with a microbrush according to manufacturer instructions, dried for 5 s and light cured for 10 s. Automix tips (Mixing Tips type 14) containing the dual-curing composite core build-up material Rebilda DC were used to coat the post and dentine surfaces. Thereafter, the corresponding dental impression of each tooth was filled with composite and placed onto the root to recreate the original crown shape. Curing was enhanced by exposing the apical margins of the restoration at the impression margins to light cure for 40 s. After 5 min, the silicone impression moulds were removed, crowns were further light cured for 40 s, trimmed, polished and the teeth were returned to the airtight vials for wet storage for one week until imaging.

Imaging and reconstruction

Prior to imaging, each sample was mounted in a PVA vial (Micro tube 2ml, Sarstedt, Nümbrecht, Germany), centrally stabilized in styropore pieces padded with moist foam to maintain a humid atmosphere in the air surrounding the samples.

Laboratory micro-CT (Laboratory μCT) (Skyscan 1172, Bruker micro CT, Kontich, Belgium) was used to image the specimens at moderate resolution (16 μm pixel size, 700 ms exposure time). Following reconstruction (NRecon 1.7.1.0, Bruker micro CT) the 3D architectures of the restorations were examined in both 2D and 3D (Fig. 1a) (ImageJ 1.52d, National Institutes of Health, Bethesda, MD, USA; CTvox, Bruker micro CT). The cervical areas, just beneath each core, were selected for high-resolution imaging in a synchrotron. Samples were scanned on beamline ID19 of the European Synchrotron Radiation Facility (ESRF, Grenoble, France) using inline propagation-based contrast enhancement. An X-ray photon energy of 34 keV with partial coherence used to minimize absorption and induce phase contrast enhancement. The imaging system consisted of a pco.edge camera (PCO AG, Kelheim, Germany), LSO scintillator in a

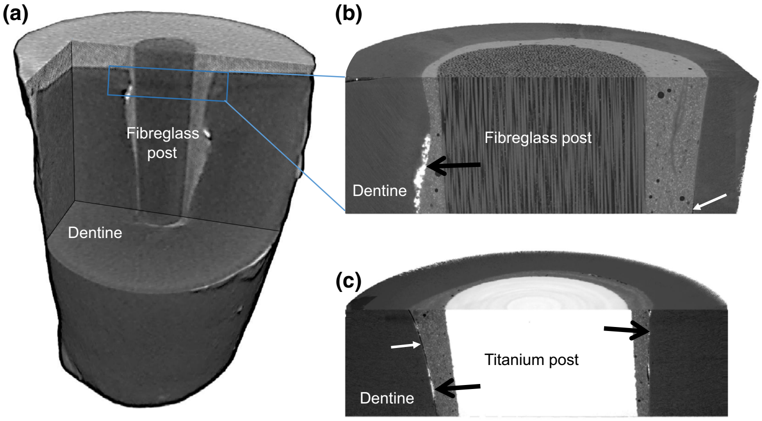


Figure 1 Low-resolution Laboratory μCT images versus high-resolution PCE-CT: (a) Laboratory μCT is useful for scans of the entire root, containing the region of interest (e.g. blue marked box) at moderate resolution. High-resolution scans by PCE-CT provided sub-micrometer resolution data of teeth restored with (b) fibre and (c) titanium posts. Note the impressive contrast and high level of detail. White arrows identify voids between dentine and cement. Black arrows show remnants of endodontic sealer.

custom-made imaging system (OptiquePeter, Lentilly, France) with an effective pixel size of 0.65 µm. PCE-CTs were obtained using a sample-detector distance of 33 mm. Each sample was mounted on the high-resolution rotation stage and in each scan, a total of 4900 radiographic projections were recorded (200 ms exposure times) whilst continuously rotating the samples by 360° (using a horizontal stitching mode to image samples wider than the field of view), requiring approximately 16 min per scan. During all stages of imaging, the samples were maintained in airtight, sealed vials maintaining saturated humidity conditions (droplets visibly condensing on the vial walls) under a constant hutch temperature of 23 °C. ESRF in-house code was used to reconstruct the data, enhancing contrast in the radiographs by means of Paganin-based filtering (delta/beta ratio of 200, Mirone *et al.* 2014).

Image processing and parameters quantification

The high-resolution reconstructed datasets were visualized using ImageJ and CTvox, and topographies typically contained more than 2100 slices along the post axis, occupying more than 80–90 Gigabytes each. Example 3D reconstructions are shown in Figs. 1 and 2 with corresponding high-resolution cross-sectional and longitudinal slices shown in Fig. 2. For data analysis, $n = 13$ artefact-free reconstructions were quantified so as to compare integrity, interface architecture, gaps and internal voids (the 14th PCE-CT scan failed due to unrecoverable data loss). The 3D datasets were reduced to produce 5 µm thick slices using the minimum intensity projection of 8 neighbouring sub-micrometer slices (Z project function of ImageJ). Slices 100 µm apart were then compared within the cervical area of each tooth, at increasing distances below the apical margins of the core build-up. Slices at identical depths of all teeth were selected for further processing for both intra- and inter-tooth comparisons. Analysis included measurements of the canal, cement and post perimeters as well as the cross-sectional areas, all performed by a single evaluator (AS). The total length of observed interfacial gaps between root and restoration materials was recorded. The length of remnants of endodontic sealer (AH Plus) on the canal perimeter was also measured. Data were used to derive four measures of quality: % canal area filled with post and with cement, % interfacial gaps and % canal perimeter covered by remnants of sealer.

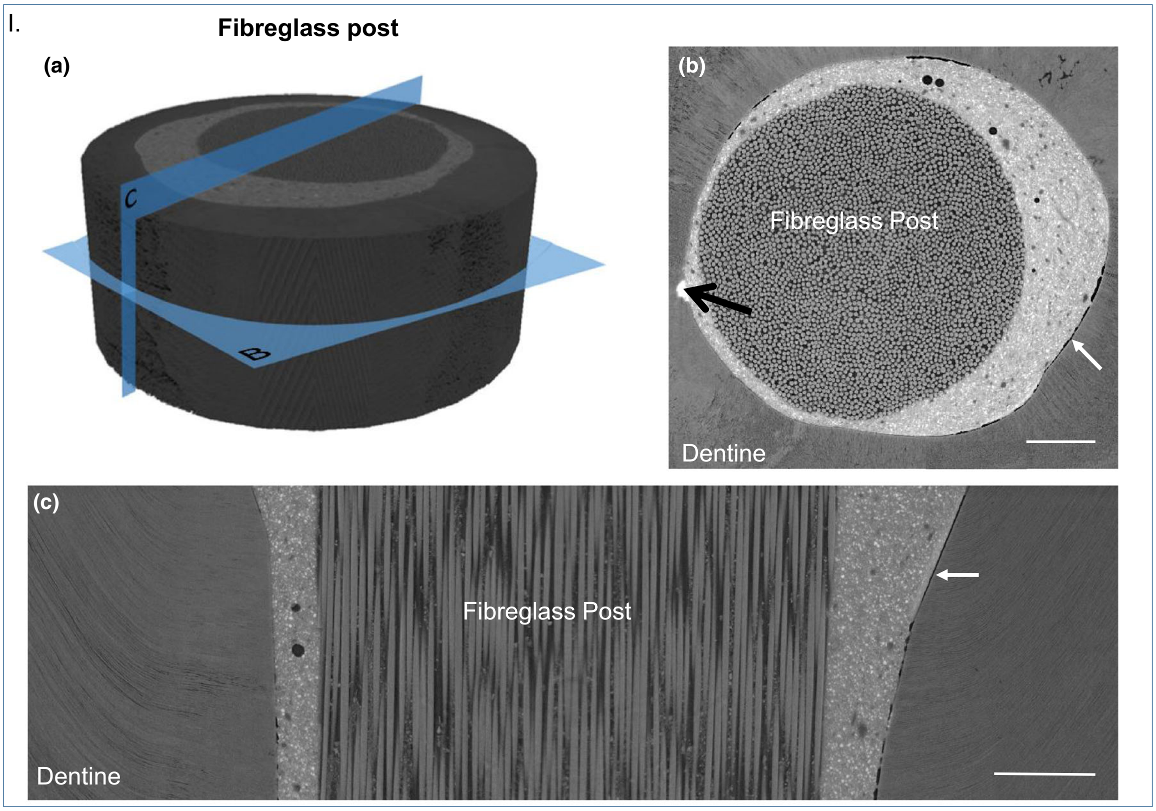
Statistical analysis

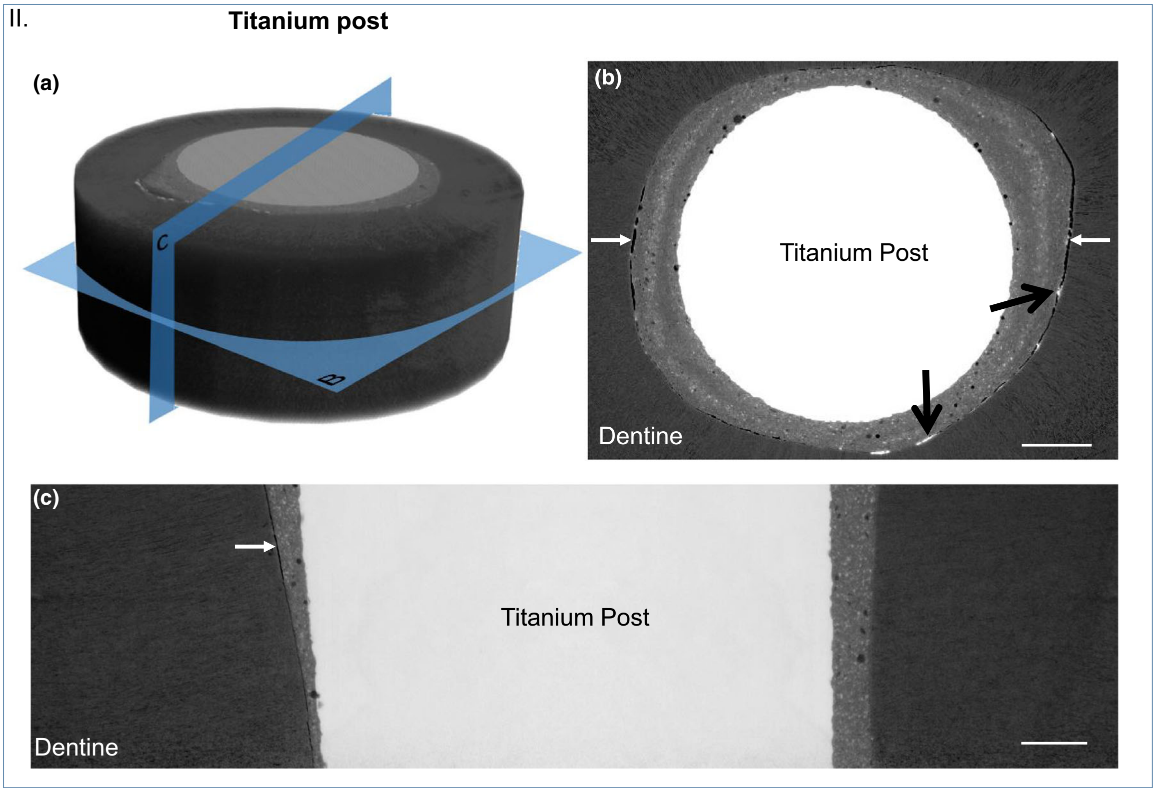
Data were tabulated and analysed using SciDAVis (version 1.23, Souceforge, Boston, MA, USA). The Mann–Whitney test was used to compare the length of interfacial gaps and percentage of interfacial gaps between groups, with significance values attributed to $P < 0.01$. The length of interfacial gaps in the uppermost and lowermost three slices of each sample was compared with a 2-way ANOVA using the *t*-Test (LSD) post hoc test. Pearson correlation coefficient (r) was determined between length of interfacial gap and different parameters (canal area, canal perimeter, cement area, post area, post perimeter and length of the perimeter with sealer remnants).

Results

Laboratory µCT reproduced all the components of the post restoration within the root, whereas the actual gaps, voids and sealer were only fully revealed by the enhanced contrast and sub-micrometer resolution of PCE-CT. As shown by 3D renderings (Fig. 1) and by virtual cross-sectional and longitudinal slices (Fig. 2), impressive details are revealed in each of the layers comprising the specimens (Videos S1 & S2). PCE-CT revealed dentinal tubules in dentine, filler particles in the cement and even single glass fibres of the fibreglass post. The high-quality images generated with PCE-CT are comparable with micrographs obtained with scanning electron microscopy (Figure S1). Importantly, at the energy of the experiment, PCE-CT has high transmission values thus undergoing little absorption by the sample. The phase contrast enhancement highlights the presence of gaps at the interfaces of self-adhesive resin cements and in other cements (Figure S2). Indeed, PCE-CT reliably detects flaws and other features at the interface between different biomaterials (Appendix S1, Cloetens *et al.* 1997) and the canal walls. In fact, the enhanced contrast highlights porosity and inclusions in pores

Figure 2 Slices derived from within the 3D data of a Fibreglass post (I) and a Titanium post (II) restored root: (a) Slices along the blue planes indicated in the 3D datasets reveal inclusions, gaps and delamination in both (b) cross-sectional and (c) longitudinal slices. White arrows identify typical interfacial gaps and black arrows point to sealer remnants.


I.
Fibreglass post
(a)
C
B
(b)
Fibreglass Post
Dentine
(c)
Fibreglass Post
Dentine


II.
Titanium post
(a)
C
B
(b)
Titanium Post
Dentine
(c)
Titanium Post
Dentine

(Figure S3). Examination of all scanned teeth showed that slices in the 3D data clearly show how the post rarely takes up a central position in the cervical area of the canal. Consequently, all slices exhibit an uneven distribution of the cement on the different sides of the post (Fig. 2, Videos S1 & S2). Sporadically, water droplets were seen, yet none of the samples had any signs of cracking of dentine, indicative that the samples maintained hydration for the entire duration of the experiment thus avoiding dehydration artefacts (Shemesh *et al.* 2018).

Geometric measurements performed on each slice along the axis of the filling reveal values for area and length estimates of the canal, filling and discontinuities at the interfaces. Average values as well as any trends in the data such as the expected taper in the cervical canal perimeter/area from the most coronal to apical region, are given in Table S1. The measurements were used to derive the percentage of the filled canal cross-section and percentage of imperfections (gaps and sealer presence) observed at the dentine–cement interfaces, as depicted in Figure 3. The results reveal expected increases in the canal areas filled by the post (Fig. 3a) and decreases in canal area filled by cement (Fig. 3b). The trend in the data and ratios of cement to canal areas with increasing depths are shown in Figure S4. Interfacial gaps were observed in ~45% of the contact area between dentine and cement revealing that discontinuities were uniformly present along the cervical canal in all of the samples (Fig. 3c). There was no significant difference ($P > 0.01$) in the % length of gaps between the three uppermost slices and the three lowermost slices of all samples, further showing the uniformity of discontinuities along the imaged canal area and between groups. No interfacial gaps were identified between cement and post in any of the samples (data not shown). At the interface between dentine and cement, many of the slices exhibited some remnants of the epoxy based endodontic sealer (AH Plus), always in intimate contact to dentine in the canal walls but not always associated with cement–dentine discontinuities. Figure 3d shows that approximately 10% of all canal perimeters both coronally and apically were contaminated with sealer, exhibiting a fairly even distribution with depth. There was a moderately positive correlation (Pearson correlation test, $r = 0.52 \sim 0.55$, $P < 0.001$) between interfacial gaps and sealer presence, and also with the canal perimeter (Figure S5), the canal area and the cement area (Table S1). No significant differences were observed in the length of interfacial gaps or in the % of interfacial gaps between groups restored with fibreglass and titanium posts (Table S1, $P > 0.01$).

Discussion

Self-adhesive resin cements are commonly employed for post cementation, but post dislodgment remains a major, poorly understood mode of failure (Rasimick *et al.* 2010). Of this group of materials, RelyX Unicem 2 has a long good track record and is widely used. The results reveal that regardless of the type of post and the depth to which it is placed, almost half of the interface surface between the cement and the root canal walls exhibited gaps. The observed micron-sized discontinuities are extremely narrow (see Fig. 2 and Figure S2), which is why they are best observed in intact teeth using reasonably high energy ($E > 30$ keV) PCE-CT. The restorations quantified in this work were performed according to manufacturer instructions and under standardized, idealized, reproducible conditions in the laboratory. They were further imaged swiftly and under fully hydrated stable conditions (watertight vials). Dehydration, contamination, incomplete adherence to treatment protocol, premature mechanical loading or patient related variability as possible causes for the observed compromised integrity of the restorations can therefore be ruled out. A closer examination of the results reveals moderately positive correlations between the presence of interfacial voids and the parameters ‘cement cross-sectional area’, ‘canal perimeter’ and ‘canal area’. However, when the interfacial gaps are normalized by the canal perimeter, there is no difference by depth as seen clearly in Figure 3c. This is noteworthy since the cement cross-sectional area decreases as the percent of canal filled with post increases (Figure S2). Consequently, the extent of interfacial gaps is not affected by the amount of cement present in the canal. Indeed Li *et al.* (2008) showed using computer simulations that the mass of resin and restoration diameter negligibly affect the stress state at the interface between dental composites and restoration margins. The mass of cement and the root canal surface dimensions must therefore be ruled out as main reasons for gap formation at the interfaces between dentine and cement during setting. The C-factor (ratio of bonded to free surface area) in these restorations is very high, as most surfaces are bonded. It is thus possible

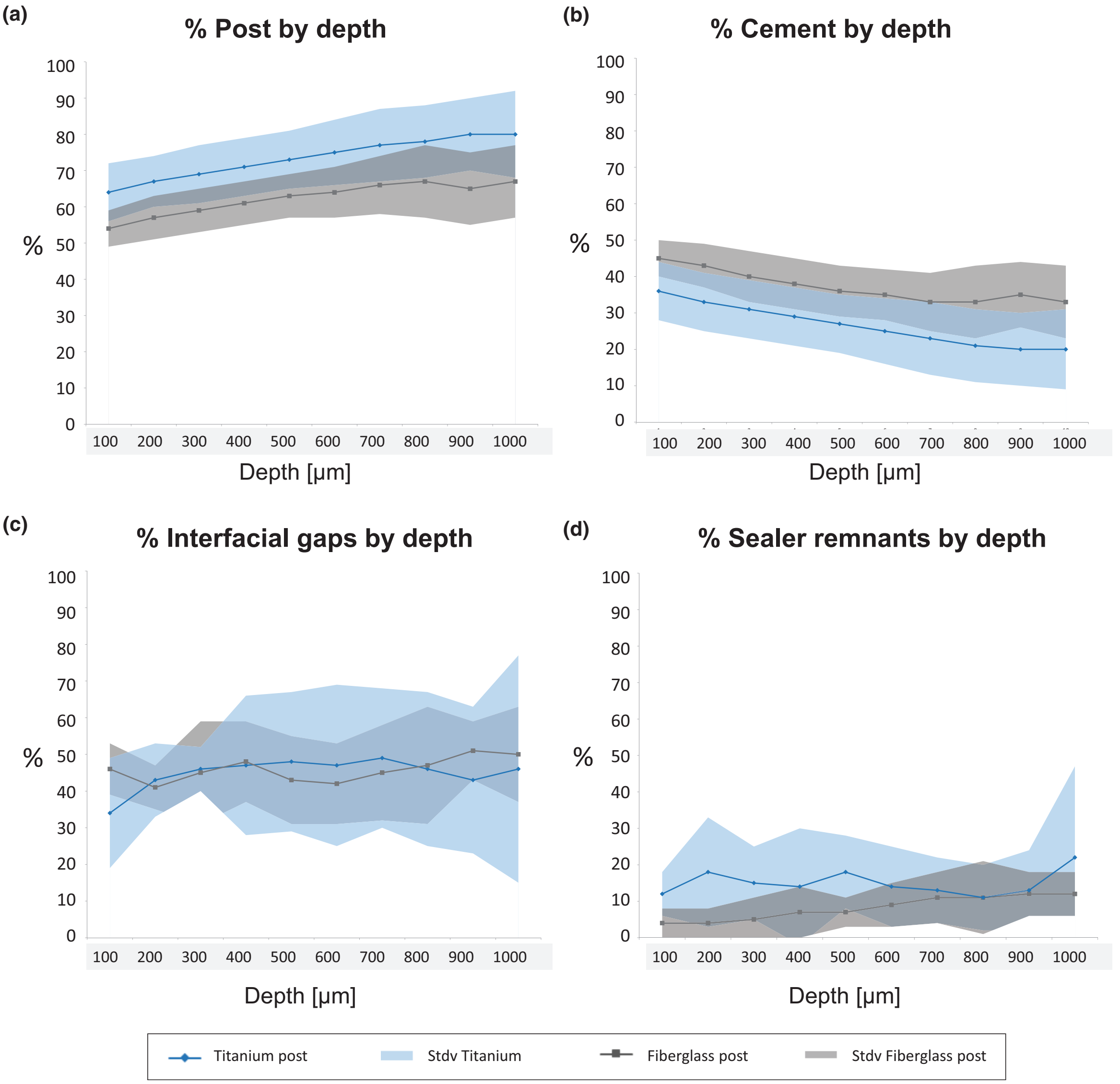


Figure 3 Trends in % filled and % gaps/sealer in titanium and fibrepost restored teeth: (a) % Post area (Post area/Canal area), (b) % Cement area (Cement area/Canal area), (c) % interfacial gaps between cement and dentine (Gaps perimeter/Canal perimeter) and (d) % remaining sealer on the perimeter of the canal walls (Sealer remnant perimeter/Canal perimeter) at increasing depths beneath the apical margin of the core buildup.

that RelyX Unicem 2 shrinkage, although low when compared to other self-adhesive resin cements (Kitzmuller *et al.* 2011), leads to stresses that develop sooner than, or are higher than the early established bond strength between the setting cement and the root dentine. Such localized stresses may then lead to widespread detachment, concentrated at the weakest interface, namely between cement and dentine along the root surface. Further work is needed to accept or refute these speculations.

The PCE-CT measurements focused on the coronal, outermost part of the cemented post, corresponding to the cervical first millimetre beneath the core build-up. The cervical tooth region exhibits reduced mass of remaining healthy dentine (due to caries or as a result of root canal treatment) and must therefore experience elevated stresses, due to tooth loading and bending during oral function. Consequently, besides resistance to bacteria and biofilm ingrowth, an important reason for the dentist to attempt to establish a defect-free cervical restoration is to create a solid structure to optimally resist mastication induced stress. The lack of homogeneous continuous interfaces between tooth and restoration questions the capacity of self-adhesive resin cements to create a so-called endodontic 'monoblock' (Tay & Pashley 2007), free of structural defects, which is essential to establish the maximal mechanical resistance to masticatory load. It is proposed that immediately after construction, thin extended gaps appear between the dentine and the resin cement at the time of placement. These then appear to remain as flaws in the constructed post-endodontic restoration. Such pre-existing cracks might progress with time either due to fatigue related to cyclic loading or following excessive overload (e.g. biting on hard bone inside soft meat) after which the post may become dislodged.

In addition to gaps, sealer remnants were observed along the canal in most samples, demonstrating that despite meticulous surface preparation and careful inspection prior to post placement, approximately 10% of the prepared canal surfaces remain 'contaminated' by the sealer used (AH Plus, Figure S3). The degree to which sealer remnants remain in the canal might be a function of the protocol used for canal preparation. Complete removal of AH Plus is extremely difficult using chemical solvents (Kuga *et al.* 2013), and therefore, it is probably best to reduce contact between the sealer and the coronal parts of the root canal. In fact, complete elimination of the sealer might require substantial removal of sound, healthy dentine, which is difficult to justify and is certain to jeopardize the root and thus the entire treatment outcome. There was only moderate correlation between gaps and the presence of endodontic sealer (Table S1). This suggests that root surface contamination, such as sealer remnants, debris from canal instrumentation, entrapped air or biofilm (not properly observable with PCE-CT imaging), might obstruct direct contact between self-adhesive cements and tooth tissues. Note, however, that 30% of the areas where there were clear signs of endodontic sealer remnants (data not shown) showed no interfacial gaps between the sealer, cement and dentine. This suggests that AH Plus does not prevent RelyX Unicem 2 from forming a gap-free contact with the sealer. In fact, previous work has clearly shown bond formation between sealer and cement (Cecchin *et al.* 2011). Overall, these findings suggest that complete removal of sealer is desirable, or better still, that new application methods/protocols are needed, designed to protect root dentine from root filling sealer contamination in coronal regions of the canal, in preparation for improving subsequent post and core construction. Additional studies are needed to address this topic.

A homogenous interface between cement and the post is necessary for the post-and-core restoration stability (Prado *et al.* 2017). Both fibreglass and titanium posts are widely used, although, due to limited studies on the subject, titanium posts are associated with greater clinical success rates (Kramer *et al.* 2019). RelyX Unicem 2 showed homogeneity of bonding to both titanium and fibreglass posts in all samples at all depths. Note that the surfaces of both types of posts were pretreated; whereas the titanium post is prefabricated with a micro-rough surface, the fibreglass posts were treated with silane immediately before cementation.

This work showcases the advantages of PCE-CT for imaging interfaces within root canal fillings (Moinzadeh *et al.* 2016). Details were revealed nondestructively and without the use of dyes, demonstrating gaps and delamination at sub-micrometer resolution. The fast scans (3 ~ 4 scans per hour) of samples mounted in controlled humid environments with minimal absorption due to the use of rather high energy (34 keV) ensure minimal radiation or dehydration damage. Consequently, interfacial voids could be measured in a truly nondestructive manner, in 3D, free of any sample preparation artefacts. Furthermore, the PCE-CT data delivers much higher resolution and contrast than that available by Laboratory μCT; hence, the use of both methods for this work delivers data that was previously not available.

The present work is not without limitations. Synchrotron-based micro tomography is not easily accessible, which limits the number of samples that can be imaged per experiment. Furthermore, in high-resolution imaging, the field of view is limited and imaging processing and reconstruction require massive computational power and processing of huge amounts of data, which restricts the regions that can realistically be investigated in a single experiment. Analysis of the 3D data is also challenging. Future artificial intelligence and machine learning approaches may pave the way to distinguish between different states of the material imaged (liquid resin, water or debris in voids) or facilitate identifying flaws in 3D as a step to improving treatment protocols.

Conclusions

The interface between RelyX Unicem 2 cemented posts and coronal root dentine was associated with gaps extending 45% of the total dentine–resin surfaces. The length of the gaps moderately correlate with the canal perimeter and the canal and cement cross-sectional areas. The percentage of interfacial gaps did not vary along the imaged canal depth, suggesting a lack of relationship between cement thickness and gap formation at the interfaces. Approximately 10% of the root canal walls revealed traces of sealer. There were no interfacial gaps between either the fibreglass or the titanium posts and cement. PCE-CT made it possible to nondestructively quantify the spatial relations and discontinuities between all phases of the moist root canal cemented post.

Acknowledgement

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Conflict of interest

Dr. Prates Soares reports funding to support her PhD from the Elsa Neumann Stipendium des Landes Berlin, making it possible to conduct the study. The other authors have stated explicitly that there are no conflicts of interest in connection with this article.

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Supporting Information

Additional Supporting Information may be found in the online version of this article:

Appendix S1. Non-destructive microscopic 3D imaging of root canal fillings by PCE-CT

Figure S1. High compatibility between both resolution and contrast observed near RelyX Unicem2 cemented titanium and fiberglass posts, imaged first by PCE-CT followed by destructive SEM imaging.

Figure S2. High-resolution PCE-CT interface gaps observed between dentin and four types of post-cementation material: (a) self-adhesive resin cement (RelyX Unicem 2), (b) resin modified glass ionomer cement (RMGIC), (c) glass ionomer cement (GIC), (d) core composite bonded with a universal adhesive system.

Figure S3. High-resolution PCE-CT reveals interface features including: traces of tubules identifiable in dentin, interfacial gaps, zones with remnants of sealer, air bubbles in the cement layer and a water droplet residing inside a large air bubble (appearing black).

Figure S4. Boxplot of the ratios between cement area and canal area along the samples (depth in μm), showcasing a decrease in the cement filled area along the canal depth.

Figure S5. Plot of the canal perimeter (mm) versus the interfacial gaps length of the canal (mm), showing a moderately positive correlation (r: 0.5493, $P < 0.01$) between both measurements.

Table S1. Means and standard deviations of each measured parameter for both groups (titanium and fiberglass posts), tabulated by depth (every 100 μm).

Video S1. Slices along the axis of a typical root canal filling restored with a fiberglass post.

Video S2. Slices along the axis of a typical root canal filling restored with a titanium post.