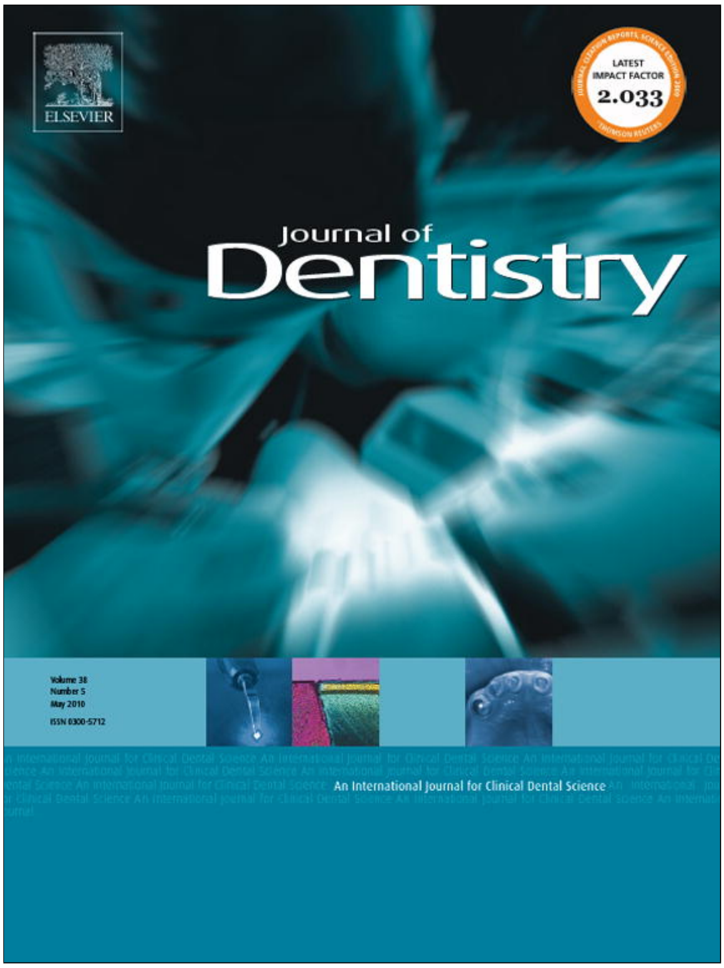
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The effect of clinically relevant thermocycling on the flexural properties of endodontic post materials

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ABSTRACT

Objectives: It is suggested that fibre-reinforced composite (FRC) posts have lower elastic moduli than metal posts and this will reduce the incidence of root fracture. However, the mechanical properties may be altered in the oral environment. The aims of this study were to determine the effect on the flexural properties of FRC and metal post materials produced by: (1) a thermocycling regime which was clinically relevant and representative of that which would occur during 1 year in the mouth and (2) storage for 1 year at body temperature.

Methods: Nine FRC and two metal post material samples were sealed in polythene sleeves and thermocycled between 10 °C and 50 °C for 10,000 cycles. Additional samples were stored dry at 37 °C for 1 year. The flexural strength and moduli were determined by three-point bending and compared with untreated control samples.

Results: Thermocycling and storage at 37 °C for 1 year decreased the mean flexural modulus of all materials. This was statistically significant for 8 of 11 materials after thermocycling, and 4 of 11 materials after storage at 37 °C ($p < 0.05$). Thermocycling and storage at 37 °C produced a non-significant increase in yield strength for both metal post materials. Thermocycling significantly increased the flexural strength of Postec while it decreased for the other FRC materials. Storage at 37 °C increased the flexural strength of three FRC materials (significantly for Postec) while it was decreased among the other materials.

Conclusions: Although some of the changes noticed in flexural properties were statistically significant, it is doubtful that they are of sufficient magnitude to affect clinical performance.

1. Introduction

Endodontic posts used in the reconstruction of severely damaged teeth have traditionally been made of metal. Over the past two decades, posts manufactured from fibre-reinforced composites (FRC) have been introduced. FRC posts exhibit comparable flexural strength to some metal posts and it has been claimed that by virtue of their lower elastic moduli, FRC posts will distribute stress within the root more favourably than their metal counterparts and consequently reduce the incidence of root fracture.[1–3] The mechanical properties of FRC materials are determined not only by the properties of the fibres and matrix resin but also by the bond between filler and matrix and by the shape, orientation and relative proportions of the reinforcing filler phase.[4,5] Endodontic posts from different manufacturers contain different matrix resins, different proportions, diameters and types of fibre and may vary in their interfacial bonding.[6] Dental restorative materials are required to maintain their properties in the challenging environment of the oral cavity where they may be in direct contact with saliva and subject to deterioration through water sorption, pH changes and enzymatic degradation. They must withstand repeated cyclic loading and the stresses induced by changes in oral temperatures. Evaluation of dental materials often includes testing of the property of interest after a sequence of thermal stressing in

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which the sample is moved between high and low temperature surroundings for a predetermined number of cycles. The temperatures selected, the duration at each temperature, the time of transfer between temperatures and the number of cycles can, and are varied by different researchers. In their review of thermal cycling, Gale and Darvell[7] concluded that the temperatures commonly chosen by investigators were too extreme to provide a representative simulation of temperature fluctuations in vivo, and suggested that 15 °C and 45 °C be used, with a transition time of 28 s at 35 °C. This is a 20 °C narrower range than the most frequently used regime of 5–55 °C as proposed in ISO 11405 recommendations (ISO, 1994). They also proposed a short dwell time of 2 s at either peak temperature. Youngson and Barclay[8] observed that in the many studies where thermocycling had been carried out, a wide range of dwell times were selected but that most employed times between 10 s and 60 s. The choice of dwell times appears arbitrary but no effect of dwell time on results has been established. Not all restorative materials however will come into direct contact with the oral cavity. Endodontic materials and posts will be enclosed by other restorative materials or root dentine. They will be insulated from dramatic temperature fluctuations,[9] but their structure may be affected by prolonged periods at body temperature. While thermocycling protocols which do not reflect clinical conditions may provide information on the behaviour of materials, prediction of their performance in the mouth would require a thermocycling regime which more closely reproduces the thermal stresses to which a restoration will be subjected in use and/or storage at body temperature for a meaningful period of time. The average number of thermal cycles which would normally occur in the mouth has been variously estimated as under 4000[10] to over 10,000 per year.[7,11]The aims of this study were thus to determine the effect on the flexural properties of FRC and metal post materials produced by (1) a thermocycling regime which was clinically relevant and representative of that which would occur during 1 year in the mouth (2) storage for 1 year at body temperature.

The null hypothesis tested was that there would be no significant effect on flexural modulus or flexural strength produced by either the chosen thermocycling regime or storage at 37 °C for 1 year.

2. Materials and methods

Samples of nine FRC and two metal materials from which posts are fabricated were supplied by manufacturers as 100 mm rods of approximately 2 mm diameter. The composition and dimensions of all the tested materials are given in Table 1.

2.1. *Measurement of flexural properties of post materials*

Each material was cut into 48 mm lengths and subjected to three-point bending on an Instron universal testing machine (Instron UK, High Wycombe, England), model 5544 with an inter-support distance of 32 mm according to ISO 3597-2. Loads were applied at 1 mm per min. The diameter of each sample was measured at six points close to the centre of the rod using a digital micrometer (Mitutoyo, Japan), and a mean calculated. Load/extension data was exported to a computer spreadsheet programme, Excel 2007 (Microsoft Corp., USA) for analysis. Ten samples of each material were tested and the flexural moduli and flexural strengths calculated using the appropriate formulae.[13] For the metal samples the yield strength was approximated by producing a 0.2% offset on the load/extension plot.

Flexural modulus of a cylindrical rod using three-point bending.[13]

$$E = \frac{4L^3}{3\pi D^4} \times \frac{F}{Y} \quad (1)$$

Table 1 – Endodontic post materials evaluated; their diameters and composition.

Material	Manufacturer	Diameter (mm)	Composition (manufacturer's information except where indicated)
Carbon fibre composites (mean fibre diameter, filler volume fraction)			
Composipost	RTD, France	1.9	Carbon 6 μm 64%; epoxy resin
Carbonite	Harald Nordin Switzerland	2.1	Carbon 6 μm 65%; epoxy resin
Glass fibre composites (mean fibre diameter, filler volume fraction)			
Aesthetiplus	RTD, France	1.9	E-glass 8 μm 62%; epoxy resin
Lightpost		2.5	Quartz glass 8 μm 60%; epoxy resin
Glassix	Harald Nordin Switzerland	2.1	E-glass 8 μm 60%; epoxy resin
Snowpost	Carbotech	2.0	E-glass with 18% Zirconia 8 μm 60%; epoxy resin
Snowlight	Ganges, France		E-glass with 18% Zirconia 8 μm 65%; polyester/methacrylate resin.
Postec	Ivoclar Schaan, Leichtenstein	2.5	E-glass 8 μm 55%; filler ytterbium trifluoride and dispersed silicon dioxide; urethane dimethacrylate/TEGMA
Easypost	Dentsply, Ballaigues, Switzerland	1.9	E-glass with 18% Zirconia 8 μm 60%; epoxy resin
Metals (alloy composition)			
Stainless Steel	Coltene/Whaledent	1.7	Fe 72.21%, Cr 18.18%, Ni 8.62%[12]
Titanium	USA	1.7	Ti 90%, Al 6%, Va 4%

Flexural strength of a cylindrical rod using three-point bending.[13]

$$\sigma = \frac{8F_m L}{\pi D^3} \quad (2)$$

where E is the flexural modulus (MPa), σ is the flexural strength (MPa), F is the applied load (N), F_m is the maximum load at break, L is the length of span between supports (mm), D is the mean diameter of the sample (mm), and F/Y is the slope of the initial linear segment of the load-deflection curve. Statistical analysis of the exported data was carried out using the computer statistical programme SPSS V16 (SPSS Inc., Chicago, USA).

2.2. *Sample thermocycling*

Ten further samples of each material underwent thermocycling to assess the effect of thermal stress on their flexural properties. Because in the clinical situation FRC posts will be surrounded by a resin cement and isolated from gross moisture within the root, all the posts were thermocycled in a dry condition. Two groups of 5 rods of each material were placed in 0.07 mm polythene sleeves, 35 mm long and 10 mm wide which were heat-sealed to prevent water ingress. A custom-built thermocycling machine moved samples between two thermostatically controlled water baths in which the sealed envelopes were immersed for 10 s. The temperatures selected for each bath were 10 °C and 50 °C with a 30 s transfer time at room temperature (23 ± 1 °C). Each sample underwent 10,000 cycles after which samples were retrieved and the flexural modulus and strengths were measured as detailed above. To measure the temperatures within the sleeves, a bead thermistor (256-045, RS components, Corby, England) was sealed into the polythene sleeve material and connected within a potential divider circuit to the input of a chart recorder. Changes in temperature produced changes in electrical output from the thermistor causing a corresponding rise or fall in the trace made on the graduated chart paper. The enclosed thermistor was placed into the water baths and underwent the same cycling regime as the posts for five complete cycles. The thermistor was calibrated in advance by placing it into five, 2 l containers of water held at different known temperatures as determined using a mercury thermometer with 0.5 °C graduations manufactured to BS1704: 1985 (ISO, 1981) (Russell Scientific Instruments, Norfolk, England) and the corresponding point was marked on the graduated chart recorder paper. The recorded numerical values were plotted against the known temperatures of the water baths. A regression line was generated with a coefficient of determination (R^2) of 0.9999 indicating a reliably linear response of the thermistor. The chart and regression line were then used to derive the temperatures being recorded during the thermal cycles.

Ten 48 mm lengths of each material were also placed in airtight polystyrene tubes and kept in an oven at a temperature of 37 °C for 1 year before undergoing three-point bend testing. Fig. 1 shows a diagrammatic representation of the experimental procedure.

An initial two-way Anova was used to test the effect of the post material and the treatment (control, thermocycling or storage at 370 °C for 1 year) on the flexural modulus and strength. For each material a one-way Anova was subsequently carried out with post hoc Scheffé tests to identify significant differences in modulus and strength between post materials untreated, thermocycled or stored at 37 °C.

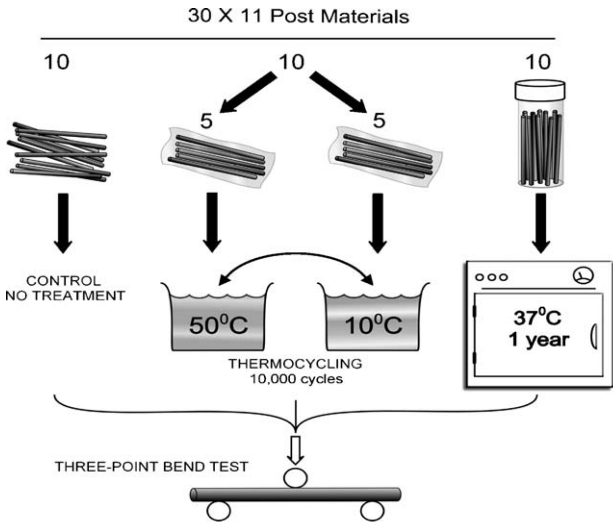

Fig. 1 – Diagram of experimental set-up for treatment of posts and three-point bend testing.

2.3. *Temperature change of samples*

The temperature changes occurring in this experimental arrangement were also compared with those that would be produced within tooth substance and restorative material. A section of the apical root of an extracted human canine tooth was shaped with a dental drill and diamond burs into a cylinder of approximately 5 ± 0.5 mm diameter and height. The root canal was enlarged to allow the bead thermistor (diameter 1.6 mm) to be inserted from one end to within 2 mm of the root apex and in contact with the root dentine which was kept moist throughout preparation. The dentine was thus approximately 1.5 mm in thickness. Both ends of the cylinder were sealed to prevent water entry using soft wax which did not extend beyond the peripheries of the opening. A cylinder of the same dimensions was made from a composite resin—Tetric (IvoclarVivadent, Schaan, Leichtenstein). The composite was packed into a 5 mm diameter transparent polystyrene tube and polymerised using a hand-held dental curing light Elipar 2500 (3M ESPE, St. Paul, USA). A channel was cut into the cylinder, again using a dental drill and the bead thermistor inserted and sealed as described above. Both cylinders were subjected to five cycles of the thermocycling regime, and the time/temperature curves recorded. The maximum and minimum mean peak temperatures were compared using independent samples t-test.

2.4. *Examination of fractured post materials*

After completion of the three-point bend testing, FRC samples were examined at 80× magnification with an incident light microscope, Model M3C (Wild, Heerbrug, Switzerland). Representative examples were also viewed under a scanning electron microscope (SEM), JEOL 5300 (Jeol Ltd, Japan) and

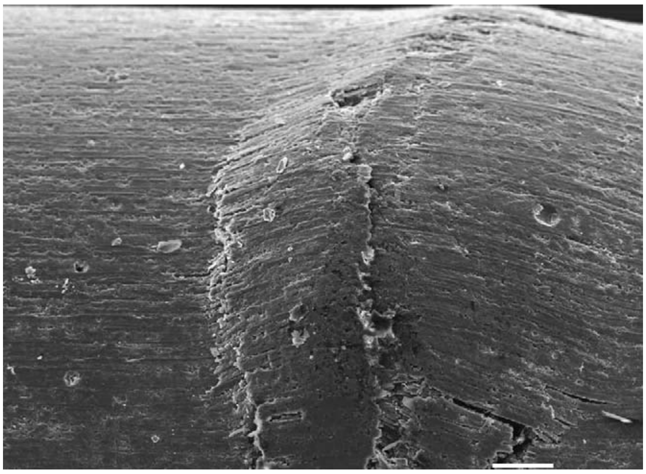

Fig. 2 – S.E.M. photograph of carbon fibre post stored at 37 °C for 1 year, after fracture in three-point bend testing showing upper compression aspect of post with buckling and rupture of fibres.

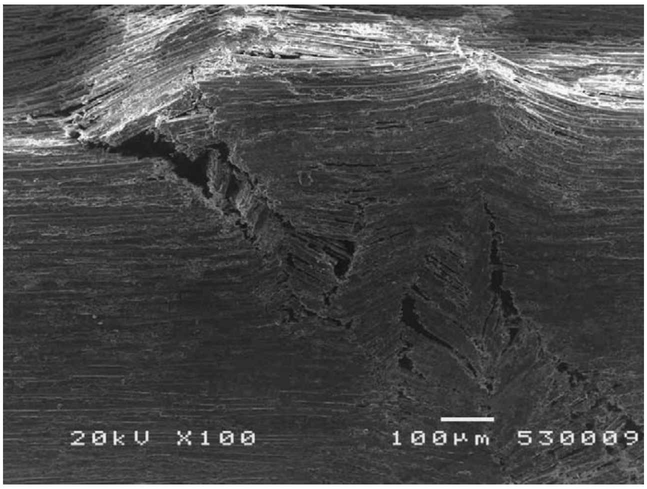

Fig. 4 – S.E.M. photograph of fractured glass fibre post showing similar appearance of broken fibres on the upper compressed surface as seen with the carbon fibre post.

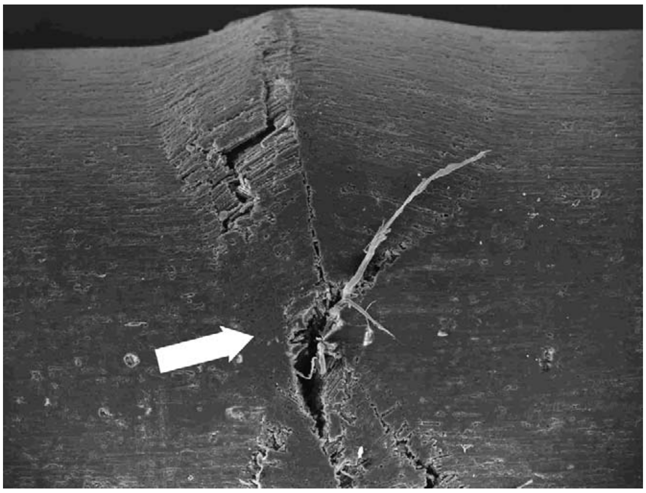

Fig. 3 – S.E.M. image of fractured carbon fibre post showing point of application of three-point bend indenter (arrowed) with buckling and splitting of fibres on either side.

images recorded. The fracture patterns seen on the upper (compression) surface were similar among the FRC materials (Figs. 2–4) as was the appearance of the lower (tension) surface (Fig. 5) No apparent differences were observed at either surface among the three treatment groups.

3. Results

The thermistor placed into a polythene sleeve recorded minimum and maximum temperatures of 13.5 °C and 48 °C. Observation of the chart recorder during the cycling process showed a delay of approximately three seconds between the thermistor/sample entering or leaving each bath and a change occurring in the temperature recording. This occurred in all three different temperature environments. No significant difference was found between the maximum ($p = 0.082$) or minimum ($p = 0.812$) mean peak temperatures recorded when the thermistor was within the cylinder of dentine (13 °C and 46 °C) or inside the composite cylinder (12.5–45.5 °C).

The flexural moduli and flexural strengths (yield strengths of metals) derived for as-received (untreated) materials, after thermocycling and after storage at 37 °C for 1 year are shown in Tables 2 and 3. After thermocycling and after storage at 37 °C, all materials showed a small decrease in mean flexural modulus. Thermocycling and storage at 37 °C increased the yield strength of both metals and the flexural strength of Snowlight, Snowpost and Postec, while it was decreased among the rest of the FRC materials.

Statistical analysis showed significant main effects for post material and thermocycling for both flexural modulus and

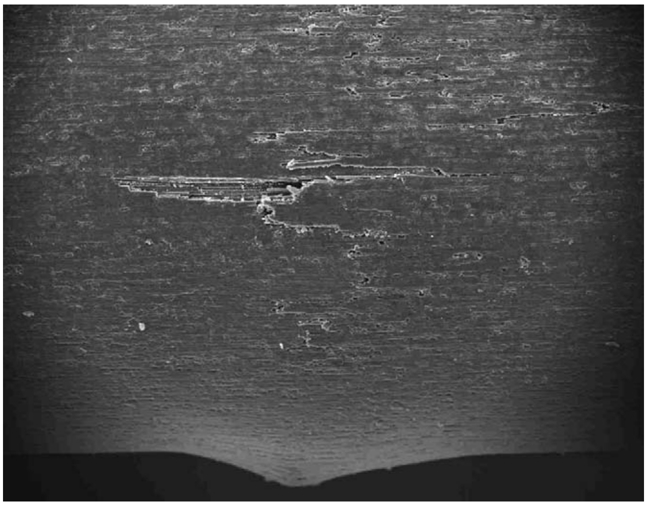

Fig. 5 – S.E.M. photograph of lower (tension) surface of thermocycled glass fibre post sample after three-point bend testing, showing a small area of fractured fibres lifting out of the surface of the post with some cracking of the matrix evident which extends peripherally.

Table 2 – Mean (S.D.) flexural moduli for each material before (untreated), after thermocycling and after storage at 37 °C.

Material	Flexural modulus GPa (S.D.)		
	Untreated	Thermocycled	37 °C
Composipost	116.90 (0.48) A	111.31 (5.65) B	113.02 (5.13) A, B
Carbonite	99.38 (1.96) A	98.44 (1.13) A	98.63 (1.24) A
Aesthetiplus	56.16 (0.63) A	54.97 (0.4) B	53.58 (0.59) C
Glassix	50.20 (1.67) A	49.05 (0.41) B	50.06 (1.64) A, B
Snowpost	45.74 (1.11) A	43.90 (1.71) B	42.94 (2.18) B
Snowlight	52.05 (1.26) A	50.46 (0.76) B	50.49 (0.79) B
Postec	44.44 (0.62) A	43.67 (0.45) B	42.66 (0.72) C
Easypost	43.96 (0.71) A	39.65 (5.77) B	42.28 (0.58) A, B
Lightpost	41.87 (0.81) A	41.37 (0.55) A	41.48 (0.92) A
Stainless Steel	193.73 (6.98) A	184.11 (9.22) B	187.3 (6.34) A, B
Titanium	115.02 (1.39) A	113.12 (3.07) A	113.95 (2.5) A

Letters in rows indicate materials with no significant differences between values for each material as determined by post hoc Scheffé tests ($p < 0.05$).

Table 3 – Mean (S.D.) flexural strengths for each material before (untreated), after thermocycling and after storage at 37 °C.

Material	Flexural strength MPa (S.D.)		
	Untreated	Thermocycled	37 °C
Composipost	1394.44 (26.35) A	1332.76 (70.04) B	1379.99 (60.85) A
Carbonite	867.91 (44.97) A	843.02 (37.95) A	856.75 (32.96) A
Aesthetiplus	1412.15 (40.47) A	1408.82 (97.7) A	1391.36 (69.78) A
Glassix	1076.43 (79.24) A	1041.83 (51.48) A	1053.54 (48.74) A
Snowpost	911.61 (43.62) A	914.30 (36.14) A	918.38 (47.92) A
Snowlight	841.70 (44.48) A	855.29 (71.24) A	849.26 (38.77) A
Postec	1215.26 (49.72) A	1267.06 (28.57) B	1322.83 (23.04) C
Easypost	949.52 (33.22) A	826.63 (158.02) B	917.53 (28.83) A, B
Lightpost	1131.08 (38.48) A	1117.17 (30.51) A	1126.85 (64.53) A
Stainless Steel	742.56 (99.35) A	782.75 (108.16) A	791.74 (66.73) A
Titanium	1477.89 (94.02) A	1527.06 (65.39) A	1534.4 (57.23) A

Letters in rows indicate no significant differences between values for each material as determined by post hoc Scheffé tests ($p < 0.05$).

strength ($p < 0.001$), and there was a significant interaction between the type of post material and whether or not it had been thermocycled ($p < 0.001$). After storage for 1 year at 37 °C, significant main effects were again identified for post material and storage at 37 °C on both flexural modulus and strength ($p < 0.001$), and also a significant interaction between the type of post material and whether or not it had been stored at 37 °C ($p < 0.001$).

The subsequent one-way Anova and post hoc Scheffé tests showed that the decrease in flexural modulus observed after thermocycling reached statistical significance for 8 of the 11 materials tested, but for only 4 out of 11 materials after storage at 37 °C. After thermocycling, a statistically significant decrease in flexural strength was recorded only for Composipost and Easypost while it was significantly increased for Postec. After storage for 1 year at 37 °C the only significant change was the increase in flexural strength observed for Postec.

4. Discussion

If the flexural properties of endodontic posts do play a dominant rôle in the performance of post-restored teeth, then it is essential that they remain unaltered by the clinical environment. In contrast to other direct restorations, prefabricated endodontic posts are separated from the oral environment within the root of the tooth by root dentine and alveolar bone; and within the crown by the core material and an indirect restoration. In this study, the thermistor recordings showed that the rate of temperature change was substantially reduced by being enclosed in composite or dentine of only 1.5 mm thickness and that the range of temperature experienced was reduced by approximately 5°. In the mouth therefore, an endodontic post is likely to be largely insulated from the effects of temperature changes occurring at the surface of a tooth. The peak temperatures reached inside the plastic sleeves were also reduced to a similar extent as those recorded within the composite and dentine cylinders, suggesting that this test arrangement provided a good simulation of the temperature gradients likely to pertain for an endodontic post within the crown of a tooth. Since thermal stress is related to the range of temperatures experienced and is increased by increasing the rate of change of heating or cooling, thermal gradients occurring in the mouth will have a greatly reduced impact on posts.

ISO 11045 suggests that thermocycling should continue for 500 cycles, but this would only correspond to the number of cycles estimated to occur in less than 2 months in the mouth. The number of cycles used in this study is representative of a more meaningful period of at least 1 year. However, despite using more clinically relevant periods of thermocycling and storage, there was little change in flexural properties observed. The significant increase in the flexural strength of the Postec samples after storage for 1 year was a surprising and unique finding. This material differs in its composition from the other FRC posts in that its matrix resin contains silicon dioxide particles and is composed of dimethacrylates rather than epoxy resins. It also has the lowest fibre content. Without further investigation it is only possible to speculate on the cause but it may be that, after being maintained at 37 °C for 1 year, the prolonged elevated temperature may have improved the interfacial bonding of the material such that flexibility was largely unaltered while fracture resistance was enhanced.

FRC posts degrade when stored in a wet environment[14,15] through plasticisation and hydrolytic degradation.[16] Thermocycling in water has been shown to weaken FRC posts and reduce their flexural modulus.[17,18] Mannocci et al. however, found no change in flexural properties of posts sealed into bovine roots and stored in water for 1 year at 37 °C.[15] This is in agreement with the results of the present study which suggest that when using a cycling and storage procedure that more closely simulates the clinical situation where water is excluded, only small changes in flexural properties of the post materials occur but that this is material dependent among the FRC materials. It would appear that the reduction in flexural properties reported by other studies may be principally the result of exposure to water rather than thermal stress as has been suggested for microleakage.[19] The effects on flexural properties were statistically significant for some materials and thus the null hypothesis is partially rejected. However, the magnitudes of the changes were small, amounting to less than 10% and are unlikely to be clinically significant. While debate continues as to the relevance of and ideal value for the elastic modulus of endodontic posts, this study suggests that, when in situ, all the endodontic post materials tested will show good consistency and stability of their mechanical properties.

5. Conclusions

The thermocycling and storage protocols employed in this study produced changes in the measured flexural properties of selected endodontic post materials. Although some of the changes noticed in flexural properties were statistically significant, it is doubtful that they are large enough to affect clinical performance.

REFERENCES

1. Qualtrough AJ, Mannocci F. Tooth-colored post systems: a review. *Operative Dentistry* 2003;**28**:86–91.
2. Duret B, Duret F, Reynaud M. Long-life physical property preservation and postendodontic rehabilitation with the Composipost. *Compendium of Continuing Education in Dentistry Supplement* 1996;**20**:S50–6.
3. Le Bell AM, Tanner J, Lassila LV, Kangasniemi I, Vallittu P. Bonding of composite resin luting cement to fiber-reinforced composite root canal posts. *Journal of Adhesive Dentistry* 2004;**6**:319–25.
4. Callister Jr WD, Rethwisch DG. Fundamentals of materials science & engineering-an integrated approach. 3rd ed. John Wiley & Sons Inc.; 2008.
5. Flinn RA, Trojan PK. Engineering materials and their application. 4th ed. John Wiley & Sons Inc.; 1994.
6. Stewardson DA. Non-metal post systems. *Dental Update* 2001;**28**:326–32. 334, 336.
7. Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. *Journal of Dentistry* 1999;**27**:89–99.
8. Youngson CC, Barclay CW. A pilot study of intraoral temperature changes. *Clinical Oral Investigations* 2000;**4**:183–9.
9. Michailesco PM, Marciano J, Grieve AR, Abadie MJ. An in vivo recording of variations in oral temperature during meals: a pilot study. *Journal of Prosthetic Dentistry* 1995;**73**:214–8.
10. Brown W, Jacobs H, Thompson R. Thermal fatigue in teeth. *Journal of Dental Research* 1972;**51**:461–7.
11. Kim JY, Takahashi Y, Kito M, Morimoto Y, Hasegawa J. Semi-quantitative analysis of early microleakage around amalgam restorations by fluorescent spectrum method: a laboratory study. *Dental Materials Journal* 1992;**11**:45–58.
12. Dionysopoulos P, Skolianos S, Papadogiannis Y. A study of the microstructure of some dental retention pins and prefabricated root canal posts. *Journal of Oral Rehabilitation* 1995;**22**:29–35.
13. Brown R. Handbook of plastics test methods. 3rd ed. Harlow, Essex: Longman Scientific & Technical; 1988.
14. Drummond JL, Toepke TR, King TJ. Thermal and cyclic loading of endodontic posts. *European Journal of Oral Science* 1999;**107**:220–4.
15. Mannocci F, Sherriff M, Watson TF. Three-point bending test of fiber posts. *Journal of Endodontics* 2001;**27**:758–61.
16. Good ML, Orr JF, Mitchell CA. In vitro study of mean loads and modes of failure of all-ceramic crowns cemented with light-cured or dual-cured luting cement, after 1 and 30 d of storage. *European Journal of Oral Science* 2008;**116**:83–8.
17. Lassila LV, Tanner J, Le Bell AM, Narva K, Vallittu PK. Flexural properties of fiber reinforced root canal posts. *Dental Materials* 2004;**20**:29–36.
18. Drummond JL, Bapna MS. Static and cyclic loading of fiber-reinforced dental resin. *Dental Materials* 2003;**19**:226–31.
19. Cenci MS, Pereira-Cenci T, Donassollo TA, Sommer L, Strapasson A, Demarco FF. Influence of thermal stress on marginal integrity of restorative materials. *Journal of Applied Oral Science* 2008;**16**:106–10.