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Dental Materials 19 (2003) 226–231

dental materials

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Static and cyclic loading of fiber-reinforced dental resin

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Received 4 July 2001; revised 6 November 2001; accepted 11 December 2001

Abstract

Objective: The aim of this study was to evaluate the flexure strength of unidirectional fiber-reinforced resins under static and cyclic loading with and without thermal cycling.

Methods: The fiber-reinforced resin materials chosen for this project were commercially available endodontic posts and commercially procured bar samples. For all materials, controls for flexure strength were tested in air and in water using three-point loading. Specimens were thermal cycled between 7 and 63 °C for 6000 cycles. A staircase approach was used to determine the flexure fatigue limit and scanning microscopy was used to examine the microstructure.

Results: The carbon/graphite fiber-reinforced resin posts and the glass FiberKor posts were significantly stronger than the ceramic (zirconia) and the other glass-reinforced resin materials. Thermal cycling caused a significant lowering (11–24%) of the flexure strength for each resin based post system. The ceramic post system decreased only by 2%. Further, for standard size glass fiber-reinforced resin bars, no significant differences between testing in air and water was observed, but a significant difference between static and cyclic loading was noted.

Significance: The decreases in the strength property due to thermal cycling and the cyclic loading of these materials indicates that their utilization in the oral environment enhances their degradation, and potentially shortens their clinical life.

Keywords: Fiber-reinforced; Flexure strength; Thermal cycling

1. Introduction

Particle strengthened dental resin composites have been used for several years for dental restorations, but are known to have limited transverse strength. It has been shown that the stiffness, strength, toughness and fatigue resistance can be improved significantly by the addition of fibers to a polymer matrix [1]. Lately, long, unidirectional fiber-containing resin materials are being introduced for a variety of dental restorative applications such as endodontic posts [2–5], splints [6], retainers, alternative to porcelain-to-metal restorations, dental bridges [7], denture bases [8,9], etc. For restorative purposes, the newer resin impregnated fiber-reinforced composites (FRC) are used for substructure construction to provide increased strength and rigidity [10–12], beneath the veneering layer of newer hybrid particulate filler composite. The intent is to provide improved esthetics and low wear characteristics, much like natural dentition. The optimal translucency of these restorations is obtained by avoiding a metal substructure framework and the metal or carbon/graphite fiber-reinforced endodontic post.

It is expected that the mechanical properties of FRC will largely be dependent on factors such as the directions of the fibers, volume fraction and aspect ratio of the fibers, impregnation of the fibers with the matrix resin, the bonding between the matrix resin and the fibers, the polymerization shrinkage of the resin, and the individual properties of the fibers and the matrix. Several studies relating these factors with the mechanical properties have been performed [13].

Several FRC products are commercially available. One product (FiberKor, Jeneric/Pentron, Inc. Wallingford, CT, USA) which contains silane coated S2 glass (Cordite) fiber in a Bis-GMA matrix was reported to have a flexure strength in the range of 453–936 MPa [11]. Such a large range in the strength was the result of variation in the fiber volume fraction, method of specimen preparation, specimen thickness and the other experimental parameters. Drummond et al. reported the flexure strength of a carbon fiber-reinforced modified epoxy resin endodontic post (C-post, Bisco Dental Products, Schaumburg, IL, USA) to be 1492 ± 19 MPa [14]. In addition, the authors also determined the pullout

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0109-5641/03/$30.00 + 0.00
PII: S0109-5641(02)00034-9

Table 1
The diameter of the products used in the investigations

Product name	Code	Diameter (mm)	Manufacturer
Carbon post	C	1.76	Bisco Dental Products, Schaumburg, IL, USA
Esthetic post	E	1.74	Bisco Dental Products, Schaumburg, IL, USA
Light post	L	1.80	Bisco Dental Products, Schaumburg, IL, USA
FiberKor post	J	1.30	Jeneric/Pentron Inc., Wallingford, CT, USA
FiberKor post	J1.25	1.09	Jeneric/Pentron Inc., Wallingford, CT, USA
FiberKor post	J1.0	0.98	Jeneric/Pentron Inc., Wallingford, CT, USA
Zirconia post	Z	1.90	Brassler Inc., Savannah, GA, USA
Luscent Anchors	LA	1.79	Dentatus USA Ltd, Lexington, NY, USA
FiberKor bars	FB	25 × 3 × 4	Jeneric/Pentron Inc., Wallingford, CT, USA

(shear) strength of several types of endodontic posts from teeth, and the influence of thermal and cyclic loading on the pullout strength [14]. An excellent review on fiber-reinforced resins for dental applications can be found in Ref. [13].

This new class of fiber-reinforced dental resin, like other restorative materials is routinely subjected to thermal cycling and cyclic loading in the oral cavity. Unfortunately for these new materials, very little attention has been paid to the interaction of these parameters with regard to strength properties, such as flexure strength and fracture toughness. The intent of this study was to evaluate the three-point flexure strength of a wide range of fiber-reinforced polymeric and ceramic endodontic posts before and after thermal cycling and to examine the effect of thermal and load cycling on the three-point flexure strength of fiber-reinforced bars (FB).

2. Materials and methods

Eight different commercial endodontic posts (Table 1) were evaluated: Carbon post (C), Esthetic post (E), and Light post (L), Zirconia post (Z), and Luscent Anchors (LA) (Dentatus, USA; Lexington, NY, USA), and three different diameter FiberKor posts (J, J1.25, J1.0). The diameter of the posts are listed in Table 1 and ranged from 1.90 to 0.98 mm. Ten posts of each group were subjected to 6000 thermal cycles between 7 and 63 °C. The thermal cycling consisted of a 30-s soak in a water bath at each respective temperature and a 5-s transfer between temperature baths. Five to ten thermal uncycled posts in each group were used as a control.

Further, FB, $25 \times 3 \times 4$ mm^3, were obtained from Jeneric/Pentron. The FBs were made from 2 mm rovings of silanated silica fiber (6–10 μm). The rovings were preimpregnated with a lightly filled (to control viscosity) resin mixture of ethoxylated Bis-GMA and polycarbonated dimethacrylate (PCDMA). The preimpregnated rovings were stacked into the rectangular Teflon mold of slightly longer size than the bar length. The mold was covered with a glass plate and pressed. This was followed by both light curing (5 min) and heat curing (15 min). Both ends of the cured bar were cut to obtain 25 mm long bar samples. On the other hand, the J posts were cut to size from a long rod drawn through a die. The rods with surface mechanical retention were made from the same kind of rovings and resin mixture as the FB bars, except that the resin was reinforced by fine silica and barium glass particles. The thermal cycling procedure for the bar samples was identical to the one used for the posts. In addition, the bars were subject to load cycling following the staircase approach as outlined by Draugn [15]. The specimens were tested in both air and water.

All the specimens were tested in three point loading on an universal materials testing system (Instron 1125, Instron Corporation, Canton, MA, USA). The loading rate was 2 mm/min with specimens supported on the jig either in air or in water. The cyclic fatigue testing was done at the same loading rate as the flexure testing using a staircase approach of either 1000 cycles or specimen fracture. This procedure would initially load the specimen to 392.28 N (40 kg), stop, lower the load to 98.07 N (10 kg), stop, return to 392.28 N (40 kg), and then continue cycling between 98.07 (10 kg) and 392.28 N (40 kg) until 1000 cycles or the failure of the specimen. If the specimen survived the 1000 cycles, the initial load for the next specimen was raised by 98.07 N (10 kg). If the specimen did not survive, but fractured before completing 1000 cycles, the initial load for the next specimen was lowered by 98.07 N (10 kg).

The flexure strength for the posts (of circular cross-section) was computed using the following equation [16]

$$\sigma = 8P_f l/(\pi d^3) \quad (1)$$

where P_f is the fracture load in N, l is the span (15 mm) between the supports, and d is the diameter (mm) of the post. For the bar samples (of rectangular cross-section), the following flexure strength formula was utilized.

$$\sigma = 3P_f l/(2bh^2) \quad (2)$$

where b is the width (mm) and h is the thickness (mm) of the specimen and l was 20 mm. For cyclic fatigue testing, the mean fatigue limit and standard deviation for each sample group was determined using the formulae and procedure outlined by Draugn [15] and Collins [17]. Statistical analysis consisted of a one way analysis of variance followed by Tukey multiple means comparison analysis.

Scanning electron micrographs (SEM) (Hitachi S3000N, Hitachi Scientific Instruments Nissei Sangyo America Ltd, Gaithersburg, MD, USA) were taken of the cross-sections of the tested samples following sputter coating of the specimen

Table 2
Flexure strength of endodontic posts with and without thermal cycling

Post-type	N[a]	Flexure strength (MPa (SD))	
C control	5	1492 (19)	a[b]
J1.0 control	10	1432 (80)	ab
J1.25 control	10	1422 (136)	ab
C thermal	10	1332 (50)	abc
J control	10	1217 (53)	bc
J1.0 thermal	5	1214 (35)	bc
Z control	10	1201 (170)	bc
Z thermal	10	1176 (182)	cd
J1.25 thermal	10	1143 (73)	cd
E control	10	1045 (57)	d
J thermal	10	963 (38)	de
L control	10	901 (82)	ef
E thermal	10	838 (89)	ef
L control	10	799 (40)	ef
LA control	10	738 (18)	g
LA thermal	10	558 (48)	h

[a] N: number of specimens tested.
[b] Mean strength joined by the same letter indicate that the values are not significantly different at $p = 0.05$ level.

surfaces with gold–palladium to delineate the microstructural differences between different product materials, and to examine qualitatively the adherence of fibers to matrix resin. All posts were mounted in one epoxy mold longitudinally and polished using the following sequences of SIC grit paper, 240, 320, 600, and 1200. By using only one mount, the cross-section of each post was examined at the same working distance and the same magnification.

3. Results

The results for the flexure strength of the endodontic posts, and the FB are presented in Tables 2 and 3. There was a wide range observed for the flexure strength of the fiber-reinforced posts. The C and J posts were the strongest. Thermal cycling caused significant decreases (11–24%) in the flexure strength of each post system. For the FB bars, which are composed of glass fiber rovings in an ethoxylated Bis-GMA/PCDMA resin matrix, the static flexure strength did not decrease when tested in water compared to air, but there was a significant lowering of strength (30–38%) when they were cyclic loaded in the respective testing conditions. Thermal cycling also resulted in a 11% decrease in flexure strength.

Table 3
The flexure strength (FS) and the flexure fatigue limit of FB

Testing condition	N[a]	Flexure strength (MPa (SD))	
Air-static	15	560 (38)	a[b]
Water-static	15	533 (36)	ab
Thermal-cyclic	10	498 (75)	b
Air-cyclic load	22	392 (32)	c
Water-cyclic load	22	330 (39)	d

[a] N: number of specimens tested.
[b] Mean strength joined by the same letter indicate that the values are not significantly different at $p = 0.05$ level.

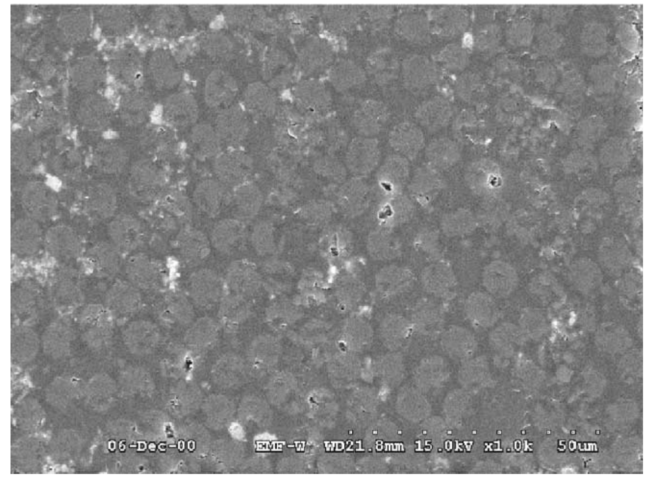

Fig. 1. Cross-section of Carbon (C) post.

Figs. 1–7 show typical SEM micrographs of cross-sections of the respective materials after being tested. The cross-section density varies between the post systems. The C post fibers are smaller in diameter (approximately 7 μm) and have a higher packing density (Fig. 1). The J posts and the E posts had a similar diameter (9–10 μm), as did the L and LA posts (13–16 μm) (Figs. 2–5). The Z post cross-section is typical of a ceramic (Fig. 6). The J posts, in addition to the glass fibers have a small particle filler incorporated into the resin between the glass fibers (Fig. 2). In addition, often irregular depression and voids on fractured fiber surfaces were seen (Figs. 2–5). Further, in all materials the interfaces between fibers and matrix were devoid of any indication suggesting de-bonding of fibers at the given magnification. The SEM micrograph of FB bar (Fig. 7) indicates the effect of hand packing.

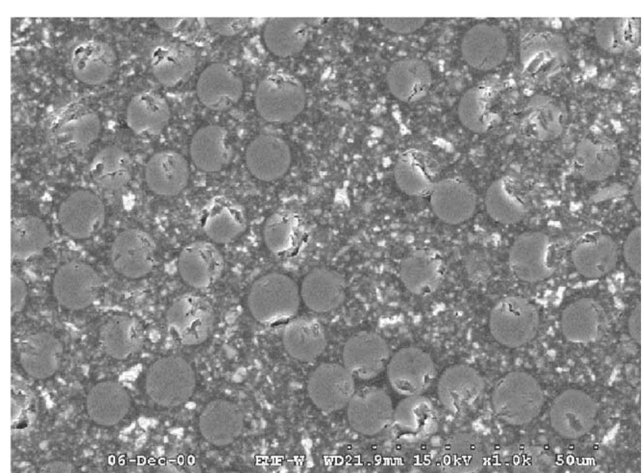

Fig. 2. Cross-section of FiberKor (J) post.

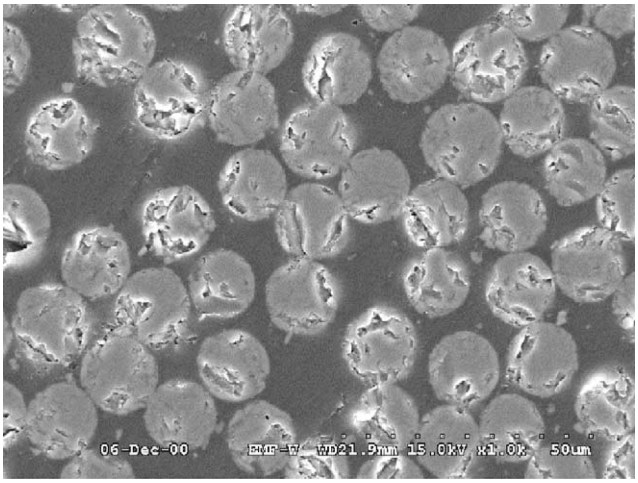

Fig. 3. Cross-section of Esthetic (E) post.

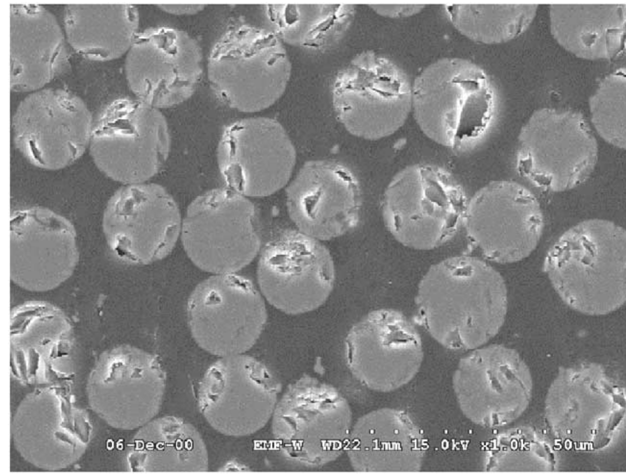

Fig. 5. Cross-section of Luscent Anchor (LA) post.

4. Discussion

In order to gain some understanding of the experimental results, it is important to know something of the compositional structural arrangement of the various fiber-reinforced post systems and their development. Frequent root fracture [18] and/or decementation, the compromising of esthetics, and the questionable biocompatibility of metallic posts created serious challenges to their continuous use [19]. The high elastic modulus of the metal post and the resulting decreased flexibility of the tooth and the poor transfer of load from post to tooth structure were other reasons for high failures of this tooth/post system. An ideal post system should have a modulus of elasticity close to that of root dentin, and the carbon fiber post fulfilled this requirement [20]. However, carbon fiber-reinforced resin matrix posts (black in color) placed serious limitations on the shade matching for the overlying restorations. In order to obtain an improved esthetic result, new carbon fiber posts were covered with quartz fiber-reinforced resin (E post) and, entire glass fiber-reinforced posts (L, J, and LA) were introduced. Also all-ceramic posts were introduced. The posts (Z posts) overcame the esthetic problem, but not the modulus of elasticity concern. The ceramic posts are very stiff and strong with no plastic behavior [21,22]. In fact, a number of studies have shown gross failures with metal or ceramic posts [23]. Further, certain all fiber-reinforced posts (E, and L) have a modified epoxy resin as a matrix capable to bond with the Bis-GMA base resin composite overlay. The J post system has a conventional Bis-GMA/PCDMA resin, which is similar to most particulate reinforced composite; and therefore, develops a continuous matrix with the overlaying material through cross-linking via a methacrylate group. This adherence is made possible by bonding agents, the presence of O_2-inhibitated surface layers, and sometimes with some products with silane coupling agents.

With respect to the flexure strength of the posts, the Z posts were the least affected by the thermal cycling (2% decrease); followed by the L and C posts (11% decrease), J1.0 posts (15% decrease), J1.25 and E posts (20%

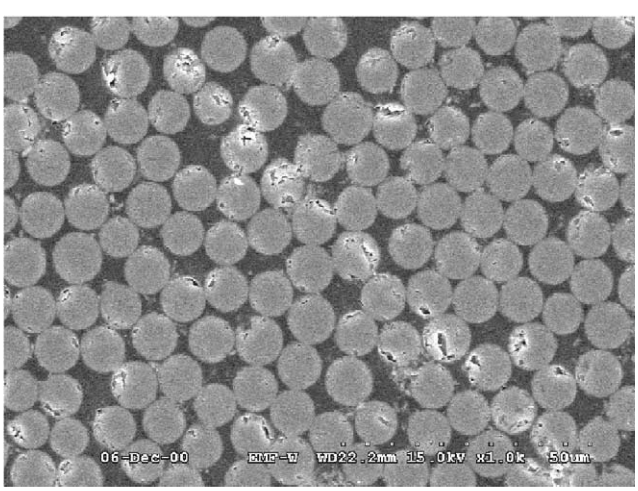

Fig. 4. Cross-section of Light (L) post.

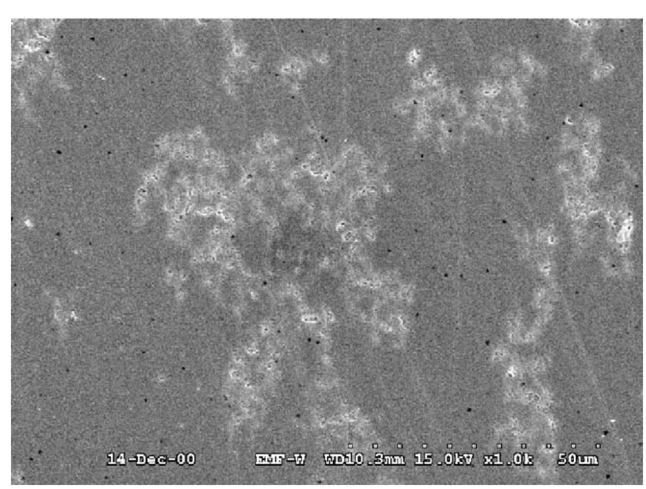

Fig. 6. Cross-section of Zirconia (Z) post.

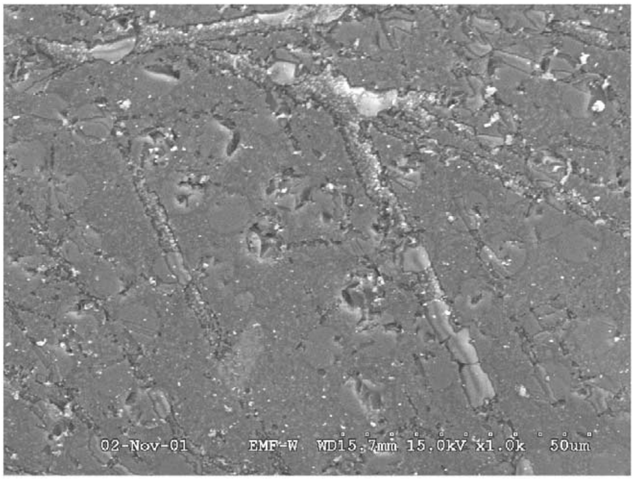

Fig. 7. Cross-section of FiberKor (FB) bar.

decrease), J posts (21% decrease), and LA posts (24% decrease). The compositional differences of the fibers may not play any role in determining different flexure strength values, since all of them have about the same elastic modulus. But, very likely the integrity of the fibers, the size, the density and distribution, and the nature of bond between matrix and the fibers which is not presently known, may be the determining factor for different flexure strength values. It would appear from the limited investigation of the variables, that even the packing density (Figs. 1–5) does not have an impact since the C post and the L post have approximately the same diameter and packing density, but the flexure strength of the L post was approximately 50% less than the C post. A study by Behr et al. found no relationship between fiber volume and flexure strength [24]. The flexure strength of fibrous posts was evaluated by Rovatti et al. [25] and found for carbon posts to be 1900 MPa and for glass posts 1200–1500 MPa, values close to those observed in this investigation.

With respect to flexure strength, all fiber post systems showed a significant lowering following thermal cycling. The thermal cycling procedure involved not only temperature changes but also immersion in water baths. Figs. 1–5 show numerous voids reflecting poor packing, and therefore, resulting in less than the optimum strength. Since the cross-sections displayed in the SEMs are from tested specimens, the cracking and destruction of the posts is partially attributed to fracture of the fibers during testing. Further, the de-bonding of fibers from matrix, and the loss of some matrix material on thermal cycling in water baths are also possibilities. Such structural changes could occur when there is degradation of the matrix resin, the bonding agent and/or the interface stress build-up due to different coefficients of thermal expansion of fiber and matrix. Present results of loss of strength on thermal cycling are consistent with the report that the immersion in water can lead to the lowering of both strength and stiffness of FRC, and it is enhanced by the increase in the water temperature [26].

The measured value of flexure strength for the J post was 1422–1492 MPa, which was more than two times higher than the FB bars, 533–560 MPa. Both the microstructure (see Figs. 2 and 7) and the processing technique for these two products as mentioned earlier, were different. Fibers in J post systems were heavily impregnated with particulate reinforced resin while in FB bars, the fibers were impregnated with lightly filled resin, though the resin composition is identical in both products. The bars are part of a laboratory system to allow the hand fabrication of fiber-reinforced resin dental appliances and then these appliances are cured, whereas the posts are a finished product as received. This hand fabrication results in fibers being lined up next to each other (Fig. 7) without resin having penetrated between the fibers, whereas, fibers in the posts (Fig. 2) were much more uniformly distributed. Other reasons for this discrepancy is that the flexure strength of unidirectional FRC depends upon the l/d (length over diameter) ratio, the loading rate and the nature of loading, and the environment of testing. A study by Behr et al. [12] found similar values for flexure strength of FB beams (585 ± 69 MPa) and a slight decrease following thermal cycling (535 ± 67 MPa) and aging in water for 30 days (499 ± 59 MPa).

The flexure strength of the FB bars was about the same magnitude when tested in air or water. This probably was the result of the impervious surface of the specimens, lack of degradation by water and/or penetration of water into the material, the enhanced adherence of resin to fibers, short duration of test and the spontaneous brittle failure of specimens. All these factors are likely to have contributed in reducing the effect of water during water testing. However, the flexure strength of FB bars was significantly lower (30–38%) for cyclic versus static loading. This is believed to be the result of repeated loading which weakens the fiber/matrix interface and/or lengthens the crack in the material to failure.

In summary, the thermal cycling and the cyclic loading caused significant decreases in the flexure strength of fiber-reinforced dental resins. Better fiber packing with improved bonding between fiber and the matrix resin is likely to improve the strength property. Also, it would appear, that the utilization of these materials in the oral cavity where thermal cycling, cyclic loading and the changing environment are constantly occurring phenomena, the degradation of the material would be enhanced with a resultant shortening of their clinical durability.

Acknowledgements

The authors would like to thank T.J. King for assistance in testing, the Research Resource Center for utilization of the electron microscope, and the manufacturers, Bisco Dental Corporation, Jeneric/Pentron, Brassler, and Dentatus for supplying the posts.

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