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Influence of triaxial braid denier on ribbon-based fiber reinforced dental composites

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ABSTRACT

Objectives. The aim of the study was to compare the mechanical characteristics of two ultrahigh molecular weight polyethylene (UHMWPE) fiber-based triaxial braided reinforcements having different denier braider yarns used in fiber reinforced dental composites to elucidate differences in response and damage under flexural loading.

Methods. Two commercially available triaxial braided reinforcing systems, differing in denier of the axial and braider yarns, using ultra high molecular weight polyethylene (UHMWPE) were used to reinforce rectangular bars towards the tensile surface which were tested in flexure. Mechanical characteristics including energy absorption were determined and results were compared based on Tukey post-test analysis and Weibull probability. Limited fatigue testing was also conducted for 100, 1000, and 10,000 cycles at a level of 75% of peak load. The effect of the braid denier on damage mechanisms was studied microscopically.

Results. The use of the triaxially braided ribbon as fiber reinforcement in the dental composite results in significant enhancement in flexural performance over that of the unreinforced dental composite (179% and 183% increase for the "thin" and "dense" braid reinforced specimens, respectively), with a fairly ductile, non-catastrophic post-peak response. With the exception of strain at peak load, there was very little difference between the performance from the two braid architectures. The intrinsic nature of the triaxial braid also results in very little decrease in flexural strength as a result of fatigue cycling at 75% of peak load. Use of the braids results in peak load levels which are substantially higher than those corresponding to points at which the dentin and unreinforced dental composites would fail. The total energy at peak load level is 56.8 and 60.7 times that at the level that dentin would fail if the reinforcement were not placed for the "thin" and "dense" reinforced braid reinforced composites, respectively.

Significance. The research shows that in addition to enhancement in flexural performance characteristics, the use of a triaxial braid provides significant damage tolerance and fatigue resistance through its characteristic architecture wherein axial fibers are uncrimped and braider yarns provide shear resistance and enable local arrest of microcracks. Further, it is demonstrated that the decrease in braider yarn denier does not have a detrimental effect, with differences in performance characteristics, being in the main, statistically insignificant. This allows use of thinner reinforcement which provides ease of placement and better bonding without loss in performance.

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1. Introduction

Fibers are increasingly being used for the reinforcement of polymer-based dental materials in prosthodontics, peridontics, and orthodontics. The introduction of fibers provides the means to directionally increase strength and stiffness, enhance fracture resistance and toughness, and decrease concerns related to creep and shrinkage. The mechanical properties of these fiber reinforced materials are intrinsically dependent on the orientation of fibers in the composite with the unidirectional configuration providing the highest strength and stiffness in the direction of the fiber. While this configuration optimizes fiber direction performance, unidirectional fiber reinforced composites have poor transverse properties resulting in the tendency for longitudinal splitting and premature failure. Further, unless the fibers are held together with transverse stitching threads (or through stitch bonding) the architecture shows significant movement during handling and application resulting in the fibers settling in a wavy and non-parallel configuration with areas that show bunching of fibers, and others which have large gaps free of reinforcement (resin-rich areas), resulting in nonuniformity of performance of the composites as well as significant deviation from the theoretical directional properties. In a number of clinical applications designed anisotropy of performance as well as ease of conformance is required. The use of bi- and multi-directional fabrics provides this tailorability, and in addition when weaves and triaxial braids are employed, the intermeshing of yarns provides integrity of fabric architecture even after the reinforcement has been manipulated to meet the configurational requirements. The architecture also provides for higher damage tolerance since resin-based damage mechanisms are essentially restricted to very small zones defined by the boundaries of the intersecting yarn directions. Details related to a range of fabric architectures can be found in Ref. [1] and will hence not be repeated herein.

In applications such as cuspal restoration multi-directional reinforcement can arrest cracks and prevent their propagation in the cervical direction, in addition to being able to redistribute the three-dimensional stress state without causing unintended debonding or fracture in the substrate or cavity. The use of tailored anisotropic reinforced composites could also aid in reducing cuspal movement noted thus far with the use of unreinforced composite resins in mesio-occluso-distal cavities in posterior teeth. In addition it is likely that these materials will enable matching of the stiffness of teeth higher than the 49% reported by Douglas [2] through the use of amalgam, and the 88% reported by Pearson and Cassin [3]. While the tailored anisotropy possible through use of different reinforcing architectures is advantageous in some application through the ability to match substrate properties, in others such as in post- and core restorations, the advantage would be in enabling designs to mimic the curve of the initial root cavity while matching the stiffness of the original root of the tooth. For example, the rigidity of currently used cast metal and ceramic posts has been noted to potentially cause root fracture [4] since the configuration and rigidity does not match that of the original root resulting in inability to evenly distribute the occlusal forces. The use of unidirectional fiber reinforcement does not enable the true mimicking of the configuration and rigidity changes, while multi-directional reinforcement could provide both the flexibility to conform to the root cavity as well as enabling matching of the original rigidity. In other applications, such as in designing fixed partial dentures care has to be taken to ensure that the components withstand masticatory loading [5] which is inherently multi-directional, and has been noted to subject inner faces of restorations to high tensile stresses which cause premature fracture initiation and failure [6]. Again multi-directional reinforcing fabrics provide the potential for tailoring configuration and response.

While these and other applications can benefit tremendously from the tailoring of reinforcement architectures in biomimetic fashion, very little work has been done to date to evaluate efficiencies of various architectures, beyond the individual testing of unidirectional, leno-weave and biaxial braided reinforcements. Although a limited number of studies have been conducted to comparatively assess the effect of various commercially available fiber-based fabric ribbons on the performance of fiber reinforced dental composites [7–10] these do not assess the actual effects of architecture, concentrating rather on global effects between brands. Recently research has, however, focused on effects of fiber orientation on thermal expansion coefficients [11,12]. A similar focus is expected to yield optimized architectures for specific dental applications both in terms of fabric conformance to the substrate configuration as well as the resulting performance characteristics. Due to exigencies of conformance and to ensure damage tolerance through encapsulation of shrinkage and microcrack initiated defects the triaxial braid architecture has significant potential. In this system fibers are generically arranged in three directions, one of which is the axial directed along the length of the reinforcing structure, and the other two, termed braiding yarns are at predetermined sets of angles (such as $\pm 30^{\circ}$ and $\pm 45^{\circ}$), with the yarns intertwined. The structure is such that no two yarns are twisted around each other and the axials are not crimped, enabling full transfer of their longitudinal reinforcing efficiency. The fabric is usually woven in the form of a flat braid providing a reinforcement ribbon of thickness equal to double the thickness of the tube wall. An advantage of this system is that there are no seams and edges, thereby enabling a higher level of integrity to be maintained during and after clinical manipulation, and negating the effects of edges that can cause higher stresses and premature damage initiation in fabric reinforced composites.

While the primary mechanical characteristics of triaxial braids in the longitudinal direction are derived from the axial yarns, aspects such as fracture toughness and strain energy can be significantly affected by the braider yarns. Conformability and ease of manipulation of fabric structures are known to decrease with increasing basis weight, especially of yarns in the off-axis direction. In a number of cases the denier of braider yarns is changed to tailor performance characteristics while preserving fiber direction strength and modulus. The objective of the current study is to investigate the effect of changes in architecture of triax-

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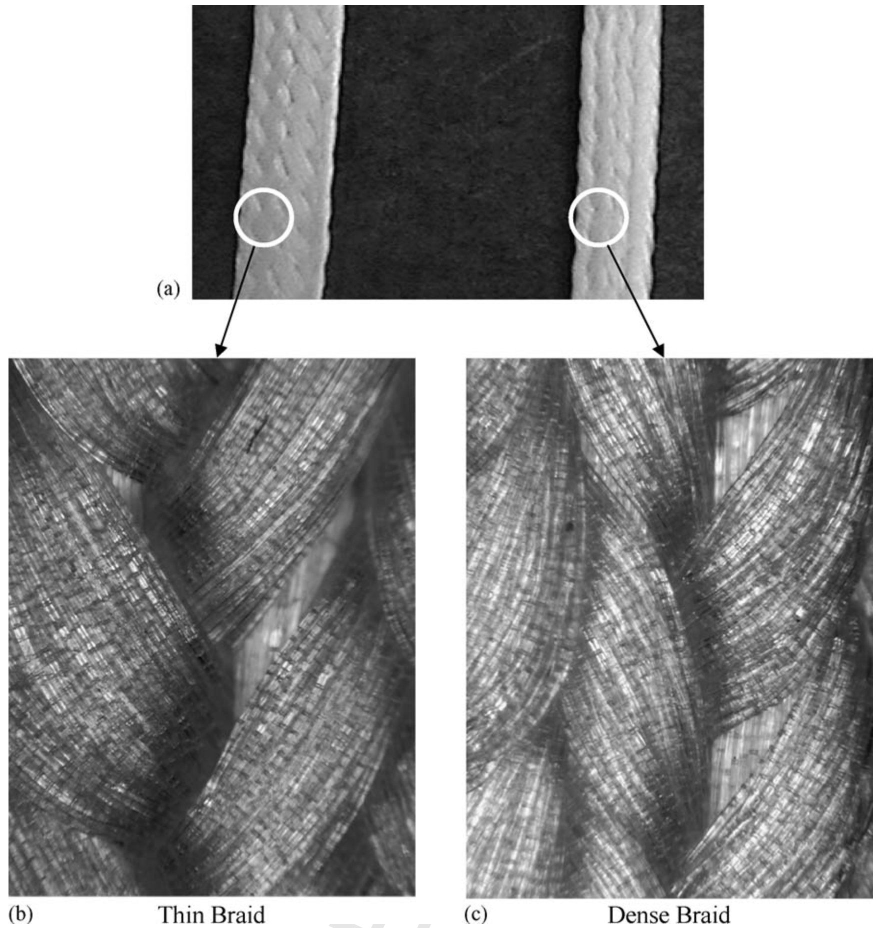

Fig. 1 – (a): Triaxial braided ribbons, with "dense" ribbon on the left and "thin" ribbon on the right, (b) micrograph of "dense" braid microstructure at 5× and (c) micrograph of "thin" braid microstructure at 5×.

ial braids, through modification of denier[1] of fibers in the braider yarns, on both flexural performance and damage initiation, and overall reliability. In the current investigation the denier of braider yarns is changed between sets resulting in two different basis weights and widths with the lower basis weight being termed the "thin" system and the higher basis weight being termed the "thick" system. The use of the thin system, which incorporates a reduction in denier of the braider yarns is an attempt to maintain the advantages of the flat triaxial ribbon while increasing ease of conformance and manipulation without substantially reducing performance characteristics of interest. In clinical applications the thinner system can be used easily both in cases where space is constrained, and when bond integrity between the tooth substrate and the restoration/reinforcement is required. It is noted that the UHMPE fibers have virtually no memory and therefore allow for ease of adaptation to the contours of teeth and dental arches. It should, however, be noted that there are still questions related to the strength of bond between these fibers and the dental composite [12] with the use of plasma treatment of fibers being reported as necessary to increase reactivity and compatibility of the fibers to ensure improved adhesion characteristics [13,14].

2. Materials and test methods

Two variations of a triaxial braided ribbon using ultra-high molecular weight polyethylene (UHMWPE) fibers obtained from Ribbond, Inc., are used as the reinforcement in this investigation. The first system incorporates 16 braider yarns at 215 denier, and is termed as the "dense" system, while the second uses 16 braider yarns at 130 denier. This system is termed as the "thin" system. Both variations use 8 axial yarns of 215 denier. Thus, the "dense" system has an effective denier of 1.65 times that of the "thin" system in the braider yarns and can thus be anticipated to have greater rigidity in that direction. The "thin" system has an overall basis weight of 2.16×10^{-4} g/mm^2, while the dense system has a basis weight of 2.34×10^{-4} g/mm^2. Fig. 1 provides a comparison of the structure of both braided ribbons. In the unmanipulated flat fabric state the thin system has a width of 2.1 mm while the dense system has a width of 2.6 mm.

[1] For purposes of clarification, denier is the unit used to specify linear density of fiber mass, with 1 denier representing a weight of 1 g per 9000 m.

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Rectangular test bars of size 40 mm length and 2.2 mm height were constructed from layered placement of a flowable composite resin (Virtuoso FloRestore, Demat), in polysiloxane molds, with glass slides held on top with rubber bands, and light cured for 60 s using a Kulzer UniXS laboratory polymerization lamp. Due to differences in width of the reinforcement ribbons the thin braids were placed within specimen widths of 2.3 mm and the dense braids were placed within specimen widths of 2.8 mm. In both cases the fabric was first wetted and then placed on the first layer of the flowable composite resin such that the fiber reinforcement was placed between 0.25 mm and 0.3 mm from the bottom surface (which would be used as the tensile surface in flexural testing). Care was taken to maintain in-plane alignment of the flat braids. Braids were noted to be well impregnated as assessed through both visual and microscopic observation. Unreinforced bars of the resin were also fabricated the same way for comparison with a nominal width of 2.3 mm.

All the specimens were tested in three-point flexure using a span of 22 mm which provides a span to depth (l/d) ratio of 10, in keeping with ISO 10477. It is noted that flexural characteristics can be substantially affected by choice of the l/d ratio which intrinsically sets the balance between shear and bending moment, with shear dominating on shorter spans. Load was introduced through a rounded cross head indenter of 2 mm diameter at a displacement rate of 1 mm/min. A minimum of five tests were conducted for each set. Loading was continued till either the specimen showed catastrophic rupture or the specimen attained a negative slope of load versus displacement with the load drop continuing slowly past peak to below 85% of the peak load.

The maximum fiber stress was determined as

$$\sigma_f = \frac{3PL}{2bd^2} \quad (1)$$

where P is the applied load (or peak load if rupture did not occur), L the span length between supports, and b and d are the width and thickness of the specimens, respectively. Flexural modulus was determined using the tangent modulus of elasticity calculated by determining the slope, m, of the initial straight-line portion of the load–deflection curve which is then used as

$$E_f = \frac{mL^3}{4bd^3} \quad (2)$$

The matrix material is generically more brittle than the fiber and usually has a lower ultimate strain. Thus, as the specimen bends the matrix is likely to develop a series of cracks with the initiation and propagation of cracks depending not just on the type and positioning of the reinforcement, but also on the strain capacity of the neat resin areas. It is thus of use to compute the strain in the composite under flexural load, and this can be determined as

$$\varepsilon_f = \frac{6dD}{L^2} \quad (3)$$

where D is the midspan displacement. The toughness of a material can be related to both its ductility and its ultimate strength. This is an important performance characteristic and is often represented in terms of strain energy, U, which represents the work done to cause a deformation. This is essentially the area under the load–deformation curve and can be calculated as

$$U = \int_0^{x_1} P\,dx \quad (4)$$

where P is the applied load and x is the deformation. In the case of the present investigation multiple levels of strain energy are calculated to enable an assessment of the response corresponding to specific strength or strain characteristics.

Intermittent and repeated cyclic loading is likely more representative of the load condition in the oral cavity than the quasi-static loading represented by the three-point flexural test. However, quasi-static tests are simpler to perform and are hence often used as the defacto-standards for the characterization of materials. In order to provide a preliminary assessment of the effect of cyclic loading on the response of the fiber reinforced composites, additional rectangular bars were loaded at a frequency of 5 Hz between a maximum load of 75% of the average peak load of the specimen type and a minimum load of 5% of the average peak load (the lower limit was set to avoid rebound of the specimen off the supports when load was reduced). The loading, introduced in sinusoidal fashion, was conducted for 100, 1000, and 100,000 cycles, after which specimens were carefully examined and then loaded quasi-statically to failure in three-point-bend.

Dynamic mechanical thermal analysis (DMTA) was used to assess glass transition temperature and viscoelastic characteristics of the sets, as well as to comparatively assess, between sets, the effect of fiber reinforcement and denier on overall characteristics. Tests were conducted in single cantilever mode using a Rheometric Scientific Model MkIII at a frequency of 1 Hz between 23 °C and 160 °C at a heating rate of 3 °C/min. For purposes of consistency the glass transition temperature (T_g) was determined from the peak of the tan δ curve, acknowledging that this temperature would be slightly higher than the more subjectively determined value from the E' curve.

3. Results

As could be expected, the application of flexural loading was seen to result in two different macroscopic forms of response as shown in Fig. 2. In the case of particulate filler dental composite specimens failure was catastrophic, in brittle fashion, after attainment of a peak load, whereas in the case of specimens reinforced with the braided ribbons the attainment of peak load was followed by a decrease in load with increasing displacement, representative of inelastic, or plastic, deformation. In these specimens failure was not through complete rupture but through a combination of flexural cracking, bridged by the UHMWPE fibers, and fiber-matrix debonding. As noted earlier the lower reactivity of the UHMWPE fibers may have contributed to the debonding mechanism. The flexural characteristics of the specimens are summarized in terms of the mean values and standard deviations in Table 1. It should be noted that since the braid reinforced specimens did not fail through rupture at the peak load level the stress at that point is

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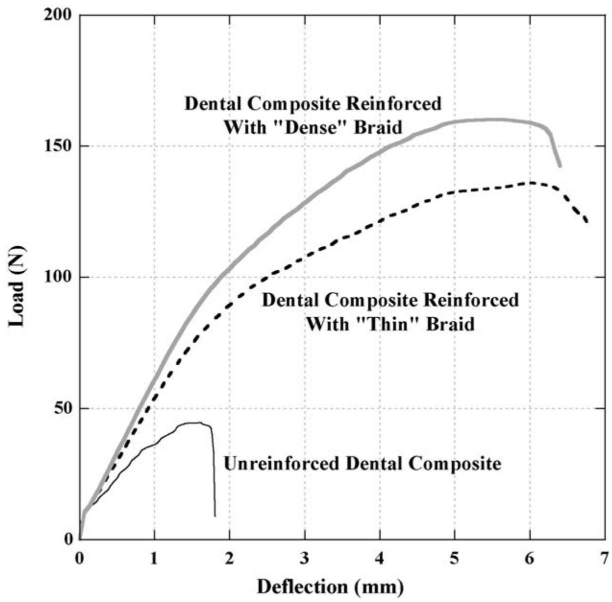

Fig. 2 – Typical load–deflection response.

termed the maximum stress. As can be seen from Table 1, the addition of the braided reinforcements significantly increases the value of all characteristics. However, with the exception of the strain determined at peak load, there is an insignificant difference in the characteristics between the two types of braided ribbon, emphasizing the dominance of the axial yarns on flexural response. The higher level of strain noted in the "thin" braid reinforced specimens is due to the greater reorientation of yarns afforded by the more open architecture of the "thin" structure, as well as the more acute angles of the braider yarns with respect to the axials (as seen in Fig. 1). In all cases, however, the addition of the braid reinforcement significantly enhances the flexural performance characteristics with the use of the "dense" ribbon resulting in slightly higher (although statistically insignificant with the exception of strain at peak load) levels of performance than the "thin" ribbon.

As seen in Fig. 2, the triaxial braided specimens did not fail by catastrophic rupture at peak load, but rather show a progressive drop in load after the attainment of the peak. As the load approaches peak flexural cracks are seen initiating from the tensile surface of the specimens and extending above the reinforcement level. The UHMWPE fibers, however, bridge these cracks holding the material together and enabling the more ductile response resulting in a gradual drop in load as the crack opening increases (essentially forming rotational hinges locally around each crack tip) with the UHMWPE fibers acting as staples till debonding takes place between the dental composite and the ribbon. The debonding, however, is seen at very high flexural strain levels.

Since the number of specimens tested in each category is small, the usual method of determination of Weibull parameters is difficult. However, following [15] the values of the Weibull shape and scale parameters, α and β, can be approximated as

$$\alpha \approx \frac{1.2}{\mathrm{COV}} \tag{5}$$

and

$$\beta = \frac{\mu}{\Gamma(1+1/\alpha)} \tag{6}$$

where COV is the coefficient of variation (determined as the standard deviation divided by the mean), μ the mean value, and Γ is the gamma function. The Weibull shape parameter for the unreinforced, "thin" and "dense" braid fiber reinforced composite materials are 32.6, 53.4 and 15.9, respectively. It is noted that a low value of the Weibull shape parameter is generally associated with broader flaw distributions and brittle materials. The corresponding scale parameters are 145.43 MPa, 399.76 MPa, and 401.72 MPa for the unreinforced, "thin" and "dense" braid reinforced materials, respectively. Since the Weibull scale parameter (its characteristic value) is defined as the value attained at 63.2% of the failure probability, the values are above those of the mean maximum flexural stress.

Cyclic loading was only conducted on a limited set of triaxial braid fiber reinforced composite specimens and very little difference was noted compared to the quasi-static loaded specimens at the three levels of cycles (100, 1000 and 10,000). It is noted that the maximum load level used in cyclic testing was only 75% of the average maximum load, and that triaxial braid architectures provide excellent fatigue resistance since any microcracks formed in the resin are arrested at the intersections of the braid unlike in unidirectional wherein the microcracks can propagate unrestricted along the fiber direction with increasing number of cycles. A minor drop in modulus of 7–10% was, however, noted in the specimens, after 100,000 cycles due to the formation of cracks initiating within the thin unreinforced layer of dental composite on the tension surface of the specimens as seen in Fig. 3. These cracks are similar to the ones seen in the quasi-static loaded specimens at load levels closer to the peak value

Use of the DMTA indicated an initial value of the storage modulus to be 2.7 GPa, 3.5 GPa and 3.6 GPa, for the unreinforced, "thin" and "dense" braid reinforced materials, respectively, with the drop in storage modulus occurring fairly early.

Table 1 – Characteristics under three-point flexure loading (values of standard deviation are shown in square brackets)

Specimen	Maximum stress (MPa)	Modulus (GPa)	Strain at peak load (mm/mm)	Energy at peak load (N mm)
Unreinforced	140.69 [10.603]	3.16 [0.036]	0.047 [0.007]	50.86 [6.798]
"Thin" braid	392.96 [14.451]	4.72 [0.453]	0.179 [0.022]	515.51 [38.954]
"Dense" braid	397.50 [8.936]	4.83 [0.263]	0.134 [0.011]	538.88 [27.794]

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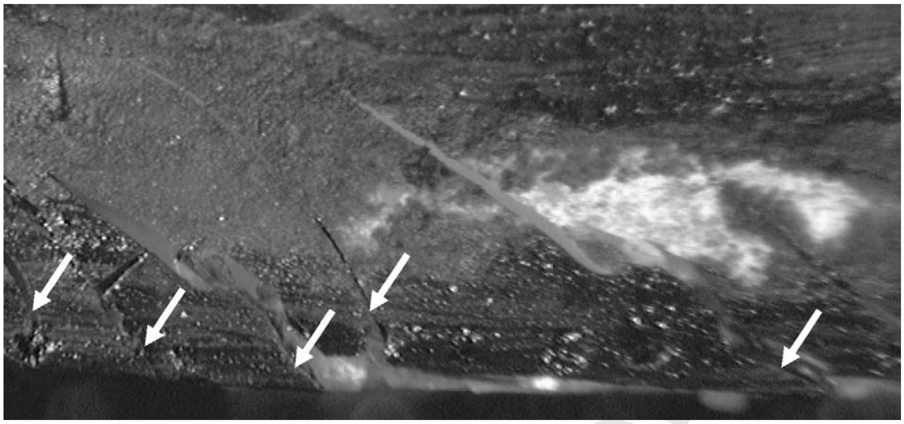

Fig. 3 – Cracking of tensile face of triaxial braid reinforced specimen.

Glass transition temperatures determined from the peaks of the tan δ curve were 89.9 °C, 94.2 °C and 95 °C for the unreinforced, "thin" and "dense" braid reinforced materials, respectively.

4. Discussion

As seen from Fig. 2 and Table 1, the addition of the triaxial braided ribbons results in significant enhancement of the flexural performance over that of the unreinforced dental composite. Using the lower of the values from the "thin" and "dense" triaxial braided specimens in comparison to the unreinforced dental resin increases of 179%, 49% and 185% are noted in maximum flexural stress, tangent flexural modulus, and strain at peak load. The addition of the triaxial braids results in significant increase in toughness (over 900% as compared to the unreinforced dental composite), as determined through the strain energy, resulting from the inherent capacity of the triaxial braid architecture to isolate and arrest cracks and defects. Further, the fabric architecture itself allows for a level of realignment, without locking of fibers, resulting in the continued absorbance of energy even after the formation of flexural cracks in the resin. Although these characteristics provide useful indicators of potential performance gains and enable comparison between specimen types, they do not provide a direct assessment of whether the material is optimized for the specific application. This is important since in a number of applications overall displacement in flexure will be required to be constrained, since excessive deformation of the bonded composite would cause failure of the substrate, and in others modulus must be tailored to ensure that the material is as close as possible to the substrate to avoid mismatch related failures. This, however, requires a more comprehensive understanding of both the base material (such as dentin and enamel) and the mechanisms of load introduction and stress transfer between various components of teeth themselves.

Two-parameter Weibull probability plots for flexural strength for the three material systems are shown in Fig. 4. The B10 levels for the unreinforced, "thin" and "dense" braid reinforced materials are 126.6 MPa, 372.9 MPa and 389.5 MPa, respectively, whereas the corresponding B50 levels are 142.1 MPa, 395.3 MPa and 399.1 MPa, respectively. It can be seen from Fig. 4 that the two triaxial braids provide approximately the same flexural stress pertaining to a given probability of failure, especially after the 60% probability level. As with the flexural performance characteristics this similarity is due to the fact that insofar as flexural response is concerned, the primary driver in the architecture is the reinforcement in the longitudinal (axial) direction, and both triaxial braids have the same number of axial yarns.

However, it must be noted that the level of performance required in an actual dental application is unlikely to reach the levels provided by the flexural test specimens, especially at the levels of bending strain noted at peak flexural load (17.9% and 13.4% for the thin and dense braid reinforced composites, respectively). It is hence of importance to assess performance of the ribbon reinforced composites at other, lower, levels. Two such levels are those related to failure of the unreinforced dental composite and the failure strain of dentin. From the current investigation the failure strain, in bending, of the unreinforced resin is noted to be 0.047. Dentin is noted to have a tensile modulus of about 18 GPa [16], and strength between 78 MPa and 91.8 MPa depending on location [17]. Using the relationship between flexure strength and tensile strength [18] with an assumed Weibull shape factor of 4.5 (which is similar to that seen in brittle foams) the equivalent flexural strength of dentin can be computed to be 228.5 MPa at the higher limit. Assuming that the flexural modulus determined from an appropriately

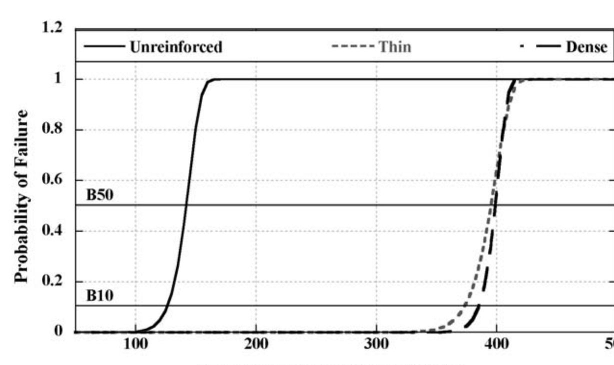

Fig. 4 – Failure probability vs. maximum flexural stress.

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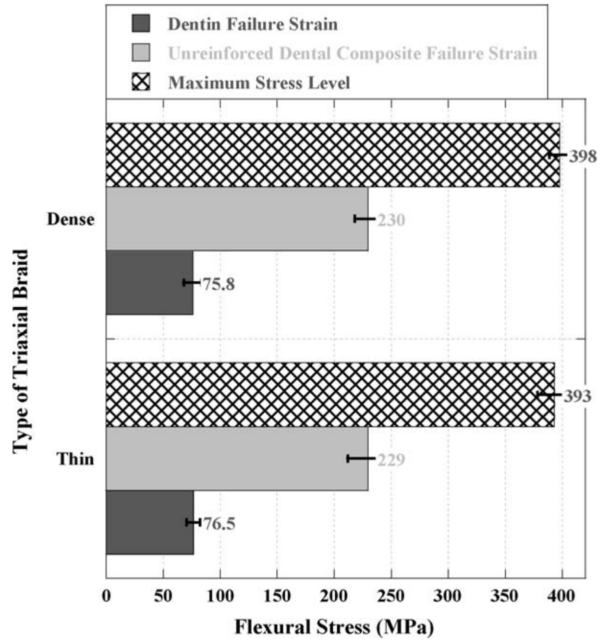

Fig. 5 – Levels of flexural stress corresponding to critical failure events.

sized flexural specimen is the same as the tensile modulus, and using the flexural strength of 228.5 MPa, a flexural failure strain of 0.013 can be assumed for the dentin. Since the fiber reinforcement in the bonded dental composite would be expected to carry load and redistribute stresses such that the dentin does not reach this level of strain, it is of interest to compare flexural characteristics of the three materials at this level, since that would enable an assessment of transfer efficiency. Comparisons of flexural stress and strain energy at the levels of dentin failure strain ($\varepsilon = 0.013$), unreinforced dental composite failure strain ($\varepsilon = 0.047$) and peak load are shown in Figs. 5 and 6, respectively. As can be seen the levels reached

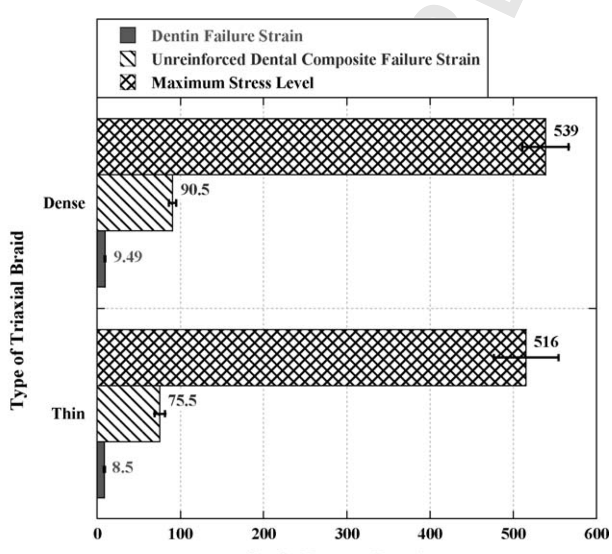

Fig. 6 – Levels of strain energy corresponding to critical failure events.

on attainment of peak load are substantially higher than those corresponding to points at which the dentin and unreinforced dental composites would fail. It is of critical note that the total energy at peak load level is 56.8 and 60.7 times that at the level that dentin would fail if the reinforcement were not placed for the "thin" and "dense" reinforced braid fiber reinforced composites, respectively.

The increase in glass transition temperature of the fiber reinforced composites over the level of the particle reinforced composites indicates that the presence of the UHMWPE fibers caused an increase in glass transition temperature. This is similar to effects of S2 glass fibers noted by Palmese et al. [19] on the cure kinetics of vinylesters. The increase in E' and T_g with increase in basis weight also indicates that the denser reinforcement, which has a higher fiber content, has a greater effect on dynamic thermal characteristics. Very small variations in E', E'' and $\tan\delta$ response were seen from tests on multiple specimens indicating good overall repeatability and stability. The use of a second run of the DMTA did not show significant change in response, again indicating good stability and attainment of a high degree of cure when specimens were fabricated.

5. Summary

It is shown that the use of triaxial braids as reinforcement significantly increases flexural characteristics of the dental composite, and that the intrinsic nature of the architecture assists in arresting cracks thereby providing a high level of fatigue resistance. A comparison of the two architectures shows that there is almost no difference in performance based on change in the number of braider yarns and that the "thin" braid provides comparable performance to the "dense" braid used conventionally. Since the thickness is less and the number of braider yarns is lower, the "thin" triaxial braid is also easier to manipulate and would conform closer to changes in substrate configuration. A comparison of characteristics with those of dentin shows that while the computed maximum flexural stress of dentin is 62.4% higher than that of the unreinforced dental composite used in this investigation, the braided composites provide over 71% enhancement in maximum stress level, emphasizing the significant strengthening capacity offered by the triaxial braided reinforcement in restorative applications. Further, the braid reinforced materials also have significantly enhanced levels of strain and energy absorption resulting in the ability to toughen fiber reinforced dental composites significantly. Further, research on bond strength and long-term integrity of these fibers is, however, recommended.

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