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Toughening of denture base resin with short deformable fibers

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ABSTRACT

Fracture resistance of polymer reinforced with short fibers consists of a sum of contributions from matrix and fiber fracture, fiber debonding and pull-out. The existing models for predicting dependence of fracture toughness on structural variables were derived for the commercially important fiber volume fractions, i.e., for $v_f \geq 0.1$. In this contribution, modification of the existing model for the dependence of the critical strain energy release rate, G_{IC} , on the fiber type, length and aspect ratio, interfacial adhesion and volume fraction has been attempted to allow predictions at low $v_f < 0.10$. The predictions based on the modified model were compared with experimental data on fracture toughness of lightly x -linked PMMA used to manufacture base of removable dentures toughened with short randomly oriented deformable fibers. The composite toughness was measured under impact loading to simulate typical mode of fracture of removable dentures. The G_{IC} for composites containing short Kevlar 29, S2-glass and poly(vinyl alcohol) (PVOH) fibers were obtained using instrumented Charpy impact tests at room temperature and impact speed of 1.0 m/s. Theoretical prediction based on the proposed model and experimental results agreed reasonably well.

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1. Introduction

Short fiber reinforced polymer composites (SFPC) constitute an important group of engineering materials combining excellent mechanical properties with reasonably easy processing. SFPC exhibit wide range of properties achieved by proper choice of structural variables and processing parameters and, hence, they can be tailored to specific applications. In majority of short fiber reinforced composites, fiber orientation is more or less random. As a result, the degree of anisotropy is generally less than in continuous fiber composites. By adding suitable fibers and by controlling factors such as aspect ratio, uniformity of the dispersion and orientation of fibers, and the fiber matrix adhesion, desired property balance can be achieved. Short fiber reinforced composites can be processed in a manner similar to the neat polymer matrix. Thus, large volume processing techniques such as injection molding can be used [1].

Poly(methyl-methacrylate) (PMMA) is a thermoplastic polymer exhibiting excellent optical properties, surface hardness and biocompatibility. In dentistry, removable dentures are one of the most widely utilized means to replace missing teeth [2]. In a removable denture, artificial teeth are embedded in an anatomically shaped denture base. Both, the artificial teeth and the denture base are made of lightly x -linked PMMA using radically polymerizing

di-methacrylates as the x -linking agents. For comfortable long lasting wear, the desired denture base material should possess a desired balance of stiffness and toughness. The stiffness of the commercial denture base materials seems satisfactory, however, the inherently low fracture toughness of PMMA is one of the major shortcomings of the PMMA based denture base resins [3–5]. Various routes have been utilized to enhance fracture toughness of both PMMA and x -linked PMMA [6–20]. In general, there are two basic strategies to enhance crack resistance in polymers. The first consists of generating controlled distribution of sub-critical defects near the crack tip to delocalize plastic deformation and the second is based on strengthening the material in bulk [21]. The first approach has been used for PMMA by blending it with acrylic rubber [22–24]. It has been shown, that rubber toughening, consisting of controlled distribution of rubbery inclusions increasing the extent of the material undergoing plastic deformation prior to fracture, has many limitations [23], namely reduced stiffness, enhanced creep and water sorption as well as increased adhesion of microbial plaque. Several attempts have been made to use the second approach by adding short glass, carbon and Kevlar fibers, however, the desired balance of mechanical properties, esthetics and biomechanics has not been achieved so far [15,20]. It has been shown [25] that the combination of the two strategies can lead to enhanced fracture resistance without compromising elastic modulus and creep.

Toughness is a complex property containing both structure related terms and geometrical variables related to the state of stress within the solid. In the case of brittle fracture with contained

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yielding, geometry independent fracture toughness can be expressed in terms of the critical strain energy release rate, G_{IC} , critical stress intensity factor, K_{IC} , while in the case of elasto-plastic failure, the critical J -integral, J_{IC} , or the critical crack tip opening displacement (CTOD), δ_{IC} , is utilized. For impact testing, the energy approach (G_{IC}/J_{IC}) seems the more appropriate, even though the current instrumented impact testing devices allow to use the critical stress intensity factor, K_{IC} , approach as well [16]. Measuring the fracture energy under impact conditions with varying notch length and properly processing the experimental data to eliminate artefacts resulting from bouncing, inertia effects and equipment vibrations, the structural interpretation of the G_{IC} or K_{IC} measured under high velocity loading can be attempted.

Widely used approach to properly calculate the critical strain energy release rate, G_{IC} , for the short fiber composite is summing the contributions from the individual fracture processes while assuming they are independent [21,26,27]. The contributing fracture processes include breaking (G_f), debonding and pulling-out the fibers (G_p) and the matrix fracture (G_m). After analyzing wide range of short fiber composites (SFC), Friedrich [26] proposed the following expression relating the structural variables to the overall critical strain energy release rate, G_{IC} , for SFC with unidirectionally aligned monodisperse short fibers

$$G_{IC} = v_{ff} \cdot \frac{5L_c \cdot \sigma_{Bf}^2}{6E_f} + v_{fp} \cdot \frac{L_c^2}{2L} \cdot \left(\frac{\sigma_{Df}}{E_f} + \frac{\sigma_{Fmax}}{6} \right) + v_m \cdot \left(\frac{1 - v_f}{v_f} \cdot d \right) \cdot \sigma_{Bm} \cdot \varepsilon_{Bm} \quad (1)$$

In Eq. (1), v_f is volume fraction of fibers, v_{ff} volume of fractured fibers, v_{fp} volume of pulled-out fibers, v_m volume fraction of matrix, L_c critical length, L fiber length, d fiber diameter, L/d aspect ratio, L/d critical aspect ratio, τ_a interfacial shear strength, σ_{Bf} fiber tensile stress, σ_{Df} necessary stress to debonding initiation, σ_{Fmax} maximum tensile strength necessary to pull-out fiber, σ_{Bm} matrix tensile strength, E_f fiber tensile modulus and ε_{Bm} matrix elongation at break, respectively.

The most significant contribution to the overall fracture energy is the energy associated with the fiber fracture. The fiber is stretched during the crack tip opening, sliding against the matrix and thus doing work. After it breaks, it retracts into the matrix releasing its stored elastic energy. These energies were calculated by Beaumont and Schultz [28] and were included in the factor

$$G_f = v_{ff} \cdot \frac{5L_c \cdot \sigma_{Bf}^2}{6E_f} \quad (2)$$

The term

$$G_p = v_{fp} \cdot \frac{L_c^2}{2L} \cdot \left(\frac{\sigma_{Df}}{E_f} + \frac{\sigma_{Fmax}}{6} \right) \quad (3)$$

accounts for the fiber debonding and pull-out during fracture [29]. Needless to say that Eqs. (1)–(3) have been derived considering linearly elastic materials. The ratio of pulled-out to fractured fibers is determined as the ratio of the critical length and the actual length of the fibers. Although several test procedures were developed to measure matrix–fiber interfacial shear strength, τ_a , the results obtained for single fiber composite are difficult to transfer into description of multi-fiber composites. Thus, matrix constrained shear strength, τ_y , can be used instead of the interfacial shear stress, τ_a , as its upper limiting value.

The contribution of the matrix to the overall fracture energy was expressed by Tetelman as [30]

$$G_m^* = v_m \left(\frac{1 - v_f}{v_f} \cdot d \right) \cdot \sigma_{Bm} \cdot \varepsilon_{Bm} \quad (4)$$

considering composite containing uniformly distributed fibers oriented in the direction perpendicular to the crack plane and assuming that the fiber have constant diameter d and constant L/d ratio and are separated by a distance D . Eq. (4) shows that the G_{Cm} increases with decreasing v_f , which may seem reasonable, reaching infinity for $v_f = 0$. According to Eq. (4), the G_{Cm} is proportional to the fiber diameter, d , which, on the other hand, seems incorrect when considering common fibers with diameter larger than 1 μm .

Fiber orientation is an important structural variable, since the fibers contribute proportionally to the projection of their length in the direction perpendicular to the crack plane. The actual short fiber orientation depends mostly on the fiber length, viscosity of the matrix and the processing route. Changes in fiber orientation are related to a number of factors, such as the geometrical properties of fibers, visco-elastic behavior of the matrix, mould design and the change in shape of the material produced by the processing operation. A correlation between $\tan \varphi$ and the fiber orientation factor, f_p , is derived as follows [26,27]:

$$f_p = 2 \cdot \langle \cos^2 \varphi \rangle - 1 \quad (5)$$

where

$$\langle \cos^2 \varphi \rangle = \frac{\sum_i N(\varphi_i) \cdot \cos^2 \varphi_i}{\sum_i N(\varphi_i)} \quad (6)$$

In Eq. (6), φ_i represents the angle between individual fiber and the load direction (perpendicular to the crack plane) and $N(\varphi_i)$ the number of fibers under certain angle φ_i . The value of f_p ranges from -1 to $+1$. The $f_p = -1$ indicates that all fibers are oriented parallel to the crack direction, whereas $f_p = 0$ corresponds to material with random fiber orientation in plane (average $\varphi = 45^\circ$). For $f_p = 1$, all fibers are aligned in the direction of loading. Thus, $\tan \varphi$ can be expressed as

$$\tan \varphi = \sqrt{\frac{1 - f_p}{1 + f_p}} \quad (7)$$

Hence, Eq. (1) can be modified to include fiber orientation

$$G_{IC} = v_{ff} \cdot \frac{5L_c \cdot \sigma_{Bf}^2}{6E_f} \cdot \left(1 - \frac{5.5\tau_{ym}}{\sigma_{Bf}} \cdot \sqrt{\frac{1 - f_p}{1 + f_p}} \right)^3 \cdot \left(1 + 0.72\varepsilon_{Bf} \cdot \frac{1 - f_p}{1 + f_p} \right) + v_{fp} \cdot \frac{L_c^2}{2L} \cdot \left(1 - \frac{5.5\tau_{ym}}{\sigma_{Bf}} \cdot \sqrt{\frac{1 - f_p}{1 + f_p}} \right)^2 \cdot \left(\frac{\sigma_{Df}}{E_f} + \frac{\sigma_{Fmax}}{6} + \frac{\sigma_{yf}}{16} \cdot \frac{(1 - f_p)^{1/2}}{1 + f_p} \right) + G_{Cm} v_m \left(\frac{1 - v_f}{v_f} \cdot d \right) \cdot \sigma_{Bm} \cdot \varepsilon_{Bm} \quad (8)$$

In Eq. (8), the τ_{ym} is the shear yield strength of the matrix, G_{Cm} is the critical strain energy release rate of the neat matrix and

$$\sigma_{Bf \max} = \frac{1}{L \cdot d} \left(\frac{1}{2} L \cdot d \cdot 2\tau_{Bi} + L \cdot d \cdot \sigma_{Bi} \right) \quad (9)$$

In this contribution, the effect of adding small amount of short deformable fibers on the fracture toughness of lightly x -linked PMMA used as denture base resin was investigated under impact loading conditions. An attempt was made to modify the existing model for the dependence of the critical strain energy release rate on the structural variables in short fiber composites to account for the effect fiber orientation and to avoid G_{IC} singularity at $v_f = 0$ in order to allow predictions for fiber volume fraction lower than 0.10. The dependence of fracture toughness for lightly x -linked PMMA modified with short deformable fibers on the fiber volume fraction was compared with predictions made using the modified theoretical model.

2. Experimental

The denture base resin used in this research was a mixture of one part of liquid methylmethacrylate (MMA) monomer and two parts of powder poly(methylmethacrylate) (PMMA) under trade name Superacryl Plus (KerrDental Prague, Czech Republic). The cross-linking has been achieved by adding 5 wt% of triethyleneglycol dimethacrylate monomer and 0.5 wt% dibenzoyl peroxide initiator into the mixture. Polyvinyl alcohol (PVAc) fibers (Kurarray, Ltd., Japan), S2-glass fibers (AGY, Inc., Belgium) or Kevlar 29 fibers (DuPont, Inc., USA), respectively, were incorporated into the resin mixture. The fiber volume fraction, v_f , varied from 0 to 0.10. Fibers were used as received without any additional surface treatment and were vacuum dried at 100 °C for 2 h prior to mixing into the resin. Properties of the materials used are listed in the Table 1.

Charpy instrumented impact tests were performed using instrumented Resil Junior impact pendulum (CEAST, Italy) at the impact speed of 1.0 m/s at room temperature and 70% relative humidity. Rectangular bars 4 mm thick (D), 6 mm wide (B) and 10 mm long (L) were heat cured in a steel mold inside a commercial laboratory pressure polymerizing chamber (TRYSTOM, CZ) at 120 °C and pressure of 0.65 MPa for 75 min. In order to determine the G_{IC} , series of specimens with starter notch of length varying from $0.1 \leq a \leq 2$ mm has been prepared. The notch tip radius was 250 μ m and was cut in the bars using automated notching device NOTCHVIS (CEAST, Italy). The G_{IC} has been obtained as a slope of the plot of fracture energy vs. the $BD\Phi$, as described elsewhere [16]. Fracture energy was measured using 4J instrumented impact hammer (CEAST, Italy) as the integral under the force–time curve.

3. Results and discussion

3.1. Model derivation

In order to eliminate the singularity of the G_{IC} for $v_f = 0$ in Eq. (8), the expression for the matrix contribution was modified assuming simplified uniform cubic spatial arrangement of fibers with constant length, L , diameter, d , and separated by a distance, D . The fiber volume fraction can be estimated as

$$v_f = \frac{\pi d^2}{4(d+D)^2} \quad (10)$$

Considering the plastic deformation in the matrix was constrained in a volume smaller than the stress intensity controlled range, it can further be assumed that the plastic deformation only

Table 1
Properties of denture base resin and the fibers used in this investigation.

Methylmethacrylate (MMA)	Molecular weight M_w	156,000
	Glass transition temperature T_g	109 °C
	Young's modulus of elasticity E_f	3.0 GPa
	Strength (static) σ_f	50 MPa
	Matrix shear strength τ_{ym}	3.45 MPa
	Strength (dynamic) σ_f	120 MPa
	Matrix elongation ε	0.1
PVOH fibers	Diameter d	14 μ m
	Aspect ratio L/d	267 and 534
	Young's modulus of elasticity E_f	36.0 GPa
	Strength σ_f	1.5 GPa
S2-glass fibers	Diameter d	13 μ m
	Aspect ratio L/d	267 and 977
	Young's modulus of elasticity E_f	89.0 GPa
	Strength σ_f	4.9 GPa
Kevlar 29 fibers	Diameter d	15 μ m
	Aspect ratio L/d	267 and 423
	Young's modulus of elasticity E_f	83 GPa
	Strength σ_f	3.6 GPa

extends into the layer of thickness D on each side of the crack plane. Then, assuming the length and width of the specimen equal to unity, the matrix contribution to fracture energy per unit area of the crack can be expressed as

$$G_{Cm} = 2D[1 - (d+D)^2 v_f^2] \int_0^{\varepsilon_{mb}} \sigma_m d\varepsilon_m \quad (11)$$

The boundary condition for the G_{Cm} should be set as

$$\lim_{v_f \rightarrow 0} G_{Cm} = 2L \int_0^{\varepsilon_{mb}} \sigma_m d\varepsilon_m = G_m \quad (12)$$

where G_m is the critical strain energy release rate of the neat matrix and the distance between fibers become equal to the ligament length. It is also assumed that fibers do not affect the matrix G_m .

Finally, it is indicated that matrix contribution to the composite toughness is directly proportional to the elastic strain energy in the matrix in the process zone near crack tip (Eq. (12)). The value of the integral shown in Eq. (12) is equal to $1/2(\sigma_{mb}\varepsilon_{mb})$ for linearly elastic matrices, assuming ε_{mb} and σ_{mb} do not change due to the presence of fibers. In the case of ideally elastic–plastic behavior of the matrix, the value of integral in Eq. (12) is equal to

$$U = \int_0^{\varepsilon_f} \sigma_m d\varepsilon = \sigma_{ym} \left(\varepsilon_{mb} - \frac{1}{2} \varepsilon_{ym} \right) \quad (13)$$

where ε_{ym} and σ_{ym} is the deformation at yield and tensile yield strength of the matrix, respectively. Thus, strain energy release rate, G_{IC} , for polymers with short deformable fibers with fiber orientation factor included can be expressed as

$$G_{IC} = v_{ff} \cdot \frac{5L_c \cdot \sigma_{Bf}^2}{6E_f} \cdot \left(1 - A \cdot \sqrt{\frac{1-f_p}{1+f_p}} \right)^3 \cdot \left(1 + 0.72\varepsilon_{Bf} \cdot \frac{1-f_p}{1+f_p} \right) + v_{fp} \cdot \frac{L_c^2}{2L} \cdot \left(1 - A \cdot \sqrt{\frac{1-f_p}{1+f_p}} \right)^2 \cdot \left(\frac{\sigma_{Df}}{E_f} + \frac{\sigma_{Fmax}}{6} + \frac{\sigma_{yf}}{16} \cdot \frac{(1-f_p)^{1/2}}{1+f_p} \right) + 2D[1 - (d+D)^2 v_f^2] \sigma_{ym} \left(\varepsilon_{mb} - \frac{1}{2} \varepsilon_{ym} \right) \quad (14)$$

3.2. Effect of fiber type on the G_{IC}

In Fig. 1, modified model for the dependence of critical strain energy release rate, G_{IC} , as a function of structural variables (Eq. (14)) is shown for PVOH fiber modified commercial denture base resin. The model predicts enhancement of the fracture toughness of about 200% adding 10 vol.% of PVOH fibers. In Fig. 2, effect of fiber type on the G_{IC} as predicted by Eq. (14) is depicted. At a given constant v_f , aspect ratio and fiber diameter, the model predicted the largest increase of G_{IC} for Kevlar 29, followed by PVOH and S2-glass fibers. In order to verify the proposed model, three types of short fibers, i.e., brittle S2-glass fibers, stiff Kevlar 29 fibers and ductile low modulus PVOH fibers, with the similar diameter and aspect ratio have been added into the commercial denture base resin (Fig. 3). At constant fiber volume fraction, $v_f = 0.1$, the Kevlar 29 fibers increased the fracture toughness of the commercial denture base resin by 250%, followed by PVOH fibers with 165% enhancement and S2-glass fibers were the least efficient toughening agent increasing G_{IC} only by 36% compared to the neat resin. Average standard deviation of less than 15% has been obtained for all the composites tested and all the measured differences were statistically significant ($p < 0.05$). Comparing the experimental data with the predictions based on Eq. (14), one can conclude that a reasonably good agreement exists between experimental data and the model predictions.

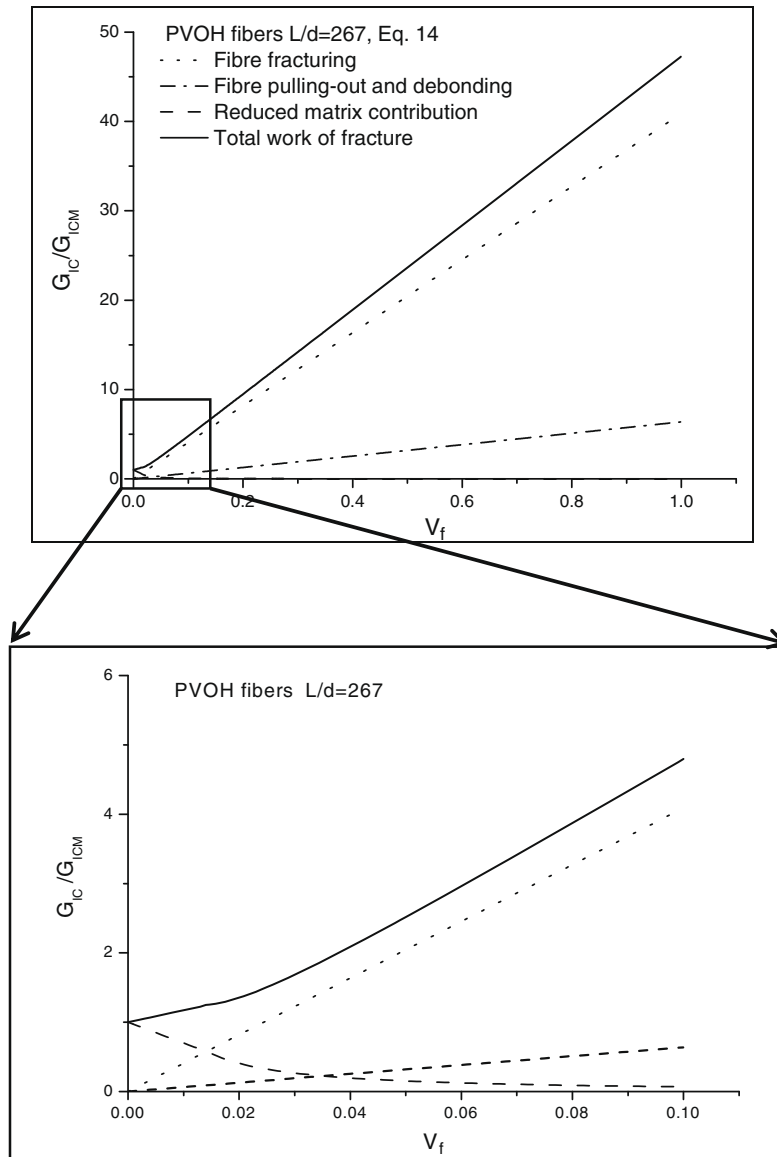


Fig. 1. The predicted concentration dependence of relative G_{IC}/G_{ICM} for the denture base resin reinforced with PVOH fibers ($L/d = 267$), calculated using Eq. (14) with modified contribution from the matrix fracture. The composite G_{IC} is equal to the matrix G_{ICM} at $v_f = 0$ as required.

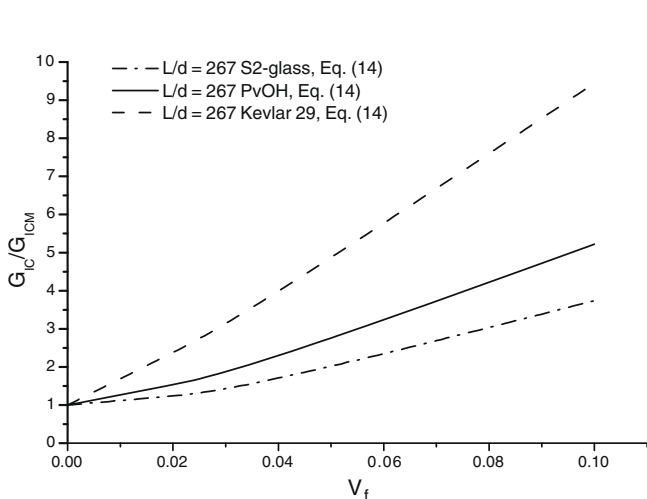


Fig. 2. The predicted effect of fiber type on the concentration dependence of the relative G_{IC}/G_{ICM} for the denture base resin reinforced with S2-glass, PVOH and Kevlar 29 fibers (initial $L/d = 267$), calculated using Eq. (14).

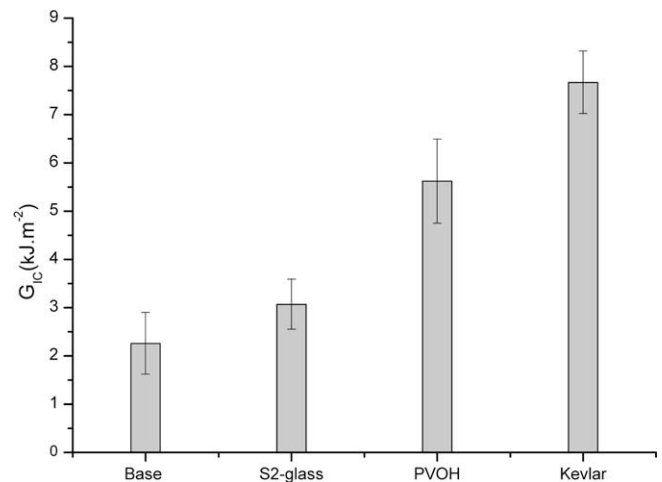


Fig. 3. The G_{IC} determined in the static test according to ASTM D366 for the denture base resin modified with S2-glass, PVOH and Kevlar 29 fibers, respectively. The $v_f = 0.10$, initial $L/d = 267$ and the tests were performed at cross head speed of 50 mm/min, at 23 °C and RH = 80%.

Table 2

Critical strain energy release rate, G_{IC} , for denture base resin modified with PVOH fibers of two aspect ratios measured under impact loading at impact speed of 1 m/s, at 23 °C and RH = 80%. Data in the table are averages and errors calculated from 10 specimens measured for each v_f and L/d ratio.

v_f	G_{IC} (kJ/m ²) ($L/d = 267$)	G_{IC} (kJ/m ²) ($L/d = 534$)
0	0.8 ± 0.1	0.8 ± 0.1
0.02	1.1 ± 0.2	0.8 ± 0.1
0.04	1.5 ± 0.2	1.0 ± 0.2
0.06	2.2 ± 0.5	1.2 ± 0.2
0.08	3.8 ± 0.7	2.2 ± 0.4
0.10	4.1 ± 0.6	2.4 ± 0.5

3.3. Effect of fiber volume fraction on the G_{IC} measured under impact loading

Adding 10 vol.% of fibers used to test the validity of Eq. (14) in the previous paragraph, however, increased the viscosity of the modified denture base dough and processing of such a material using standard dough technique in a dental laboratory would not be possible. In addition, low toughening efficiency of S2-glass fibers and yellow color of the Kevlar 29 fibers eliminated these two fiber types from further investigation due to stringent medical and esthetic requirements imposed on dental materials. Hence, in order to evaluate the efficiency of the toughening effect of adding short deformable translucent fibers into a brittle matrix, the effect of the PVOH fiber aspect ratio on the G_{IC} was investigated over the interval of v_f ranging from 0 to 0.1. Two different fiber aspect ratios ($L/d = 267$ and $L/d = 534$) were used and the experimental data were compared with predictions based on Eq. (14). In Fig. 4, values of G_{IC} for PVOH/denture base composite relative to G_{ICm} for neat denture base resin were plotted as a function of fiber volume fraction for two fiber aspect ratios. The G_{IC} obtained for the composites with shorter PVOH fibers was always greater than that for the denture base containing longer fibers (Table 2). Morphological observations of the fracture surfaces performed using scanning electron microscopy (SEM) and confocal laser scanning microscopy (CSLM) revealed more uniform distribution of shorter fibers compared to longer fibers. Moreover, some of the longer fibers embed-

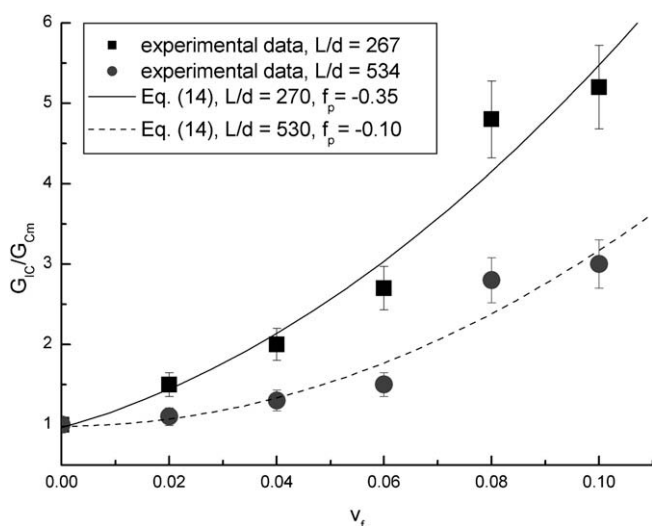


Fig. 4. Comparison of the relative G_{IC}/G_{ICm} for the denture base resin modified with PVOH fibers of two aspect ratios (initial $L/d = 267$ and $L/d = 534$) calculated using Eq. (14) with experimental data. Fiber orientation factor, f_p , has been determined from the polished SEM photographs of the fracture surfaces using the image analysis software (HarFA). The $f_p = -0.35$ was determined for $L/d = 267$, and the $f_p = -0.1$ was determined for $L/d = 534$. Tests were performed at impact speed of 1 m/s, at 23 °C and RH = 80%.

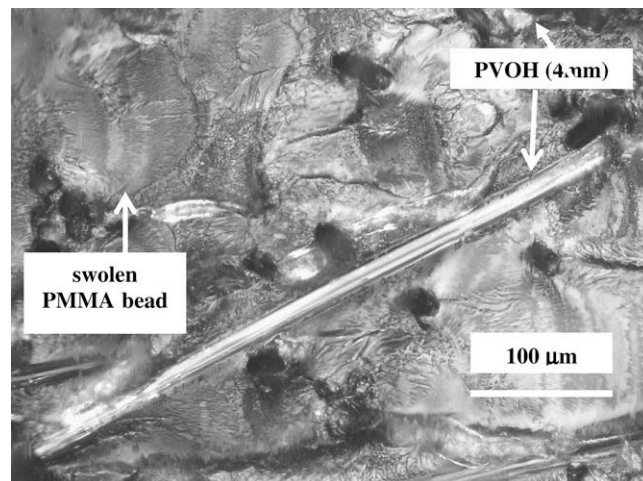


Fig. 5. Morphology of the short PVOH modified denture base resin as revealed by the laser scanning confocal microscopy. The “dough” technology used to process the mixture of PMMA beads with the MMA monomer liquid produces heterogeneous morphology consisting of swollen beads embedded in a PMMA continuum. These swollen beads possess different properties and cause stress concentration in the matrix resulting in multiple crack initiations.

ded in the denture base resin were bent, hence, their reinforcing and toughening efficiency was greatly reduced (Fig. 5). Taking into account differences in the orientation factor, G_{IC} predicted using Eq. (14) has agreed reasonably well with the experimentally measured G_{IC} , over the entire interval of fiber volume fractions used.

4. Conclusions

Effect of adding small amount of short deformable fibers into lightly cross/linked PMMA, used as denture base resin, on its fracture toughness was investigated. Predictive model proposed previously by Friedrich for the fracture toughness of short fiber reinforced composites was slightly modified in respect to the contribution of the matrix fracture to the overall critical strain energy release rate, G_{IC} , of the composite containing small fiber volume fraction. Even though the fiber pull-out and debonding are the main contributions to the G_{IC} , fiber orientation and interfacial adhesion play also a significant role. Unlike for the brittle S2-glass fibers, in the case of more ductile PVOH and Kevlar fibers, contribution from crack bridging and fiber deformation became very important. It has also been found that adding 4 mm fibers resulted in significantly greater enhancement of composite fracture toughness compared to the 8 mm fibers, at the same v_f . This has been attributed to more uniform distribution and straightness of the shorter fibers. Although very structural parameter sensitive, the theoretical results and experimental data agreed reasonably well.

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