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Analysis of Fracture and Toughness of a Denture Base with Polyvinyl Fibers

E.N. Ganesh

***Abstract:** Fracture resistance of polymer reinforced with short fibers consists of a sum of contributions from matrix and fiber fracture, fiber de-bonding 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.*

***Keywords:** Fracture Toughness, Impact Test, Short Fiber Composite, Denture Base Resin.*

I. 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 yielding, geometry independent fracture toughness can be expressed in terms of the critical strain energy release rate, GIC, critical stress intensity factor, KIC, while in the case of elasto-plastic failure, the critical J-integral, JIC, or the critical crack tip opening displacement (CTOD), □IC, is utilized. For impact testing, the energy approach (GIC, JIC) seems the more appropriate, even though the current instrumented impact testing devices allow to use the critical stress intensity factor, KIC, approach as well [16].

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***** Correspondence Author
Dr. E.N. Ganesh*, Dean, Department of Electronics & Communication Engineering, Vels University, Chennai, (Tamil Nadu), India.

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Measuring the fracture energy under impact conditions with varying notch length and properly processing the experimental data to eliminate arte-facts resulting from bouncing, inertia effects and equipment vibrations, the structural interpretation of the GIC or KIC measured under high velocity loading can be attempted. Widely used approach to properly calculate the critical strain energy release rate, GIC, 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 (Gf), debonding and pulling-out the fibers (Gp) and the matrix fracture (Gm). After analyzing wide range of short fiber composites (SFC), Friedrich [26] proposed an expression relating the structural variables to the overall critical strain energy release rate, GIC, for SFC with unidirectionally aligned monodisperse short fibers. This model has been modified by Jancar et al [28]:

$$G_{Ic} = v_{ff} \cdot \frac{5L_c \cdot \sigma^2_{Bf}}{6E_f} \cdot \left(1 - A \cdot \sqrt{\frac{1-f_p}{1+f_p}}\right)^3 \cdot (1 + 0{,}72\varepsilon_{Bf} \cdot \frac{1-f_p}{1+f_p}) +$$
$$+ v_{fp} \cdot \frac{L_c^2}{2L} \cdot \left(1 - A \cdot \sqrt{\frac{1-f_p}{1+f_p}}\right)^2 \cdot \left(\frac{\sigma_{Df}}{E_f} + \frac{\sigma_{F\max}}{6} + \frac{\sigma_{yf}}{16} \cdot \frac{(1-f_p)^2}{1+f_p}\right)^{\frac{1}{2}} + \quad (1)$$
$$+ 2D\left[1 - (d+D)^2 v_f^2\right]\sigma_{ym}\left(\varepsilon_{mb} - \frac{1}{2}\varepsilon_{ym}\right)$$

In Eq.(1), vf is volume fraction of fibers, vff volume of fractured fibers, vfp volume of pulled-out fibers, vm volume fraction of matrix, Lc critical length, L fiber length, d fiber diameter, L/d aspect ratio, Lc/d critical aspect ratio, □a interfacial shear strength, σBf fiber tensile stress, σDf necessary stress to debonding initiation, σFmax maximum tensile strength necessary to pull-out fiber, σBm matrix tensile strength , Ef fiber tensile modulus and εBm matrix elongation at break, respectively. Fiber orientation factor, fp, can be expressed as [26, 27]:

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

The value of fp ranges from -1 to +1. The fp = -1 indicates that all fibers are oriented parallel to the crack direction, whereas fp = 0 corresponds to material with random fiber orientation in plane (average φ = 45°). For fp = 1, all fibers are aligned in the direction of loading. 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 GIC singularity at vf = 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.

II. EXPERIMENT

The denture base resin used in this research was a mixture of 1 part of liquid methylmethacrylate (MMA) monomer and 2 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, vf, varied from 0 to 0.10. Fibers were used as received without any additional surface treatment and were vacuum dried at 100 oC for 2 hours 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 4mm thick (D), 6 mm wide (B) and 10mm long (L) were heat cured in a steel mold inside a commercial laboratory pressure polymerizing chamber (TRYSTOM, CZ) at 120 oC and pressure of 0.65 MPa for 75 min. In order to determine the GIC, series of specimens with starter notch of length varying from 0.1 ≤ a ≤ 2 mm has been prepared. The notch tip radius was 250 □m and was cut in the bars using automated notching device NOTCHVIS (CEAST, Italy). The GIC has been obtained as a slope of the plot of fracture energy vs. the BD□, as described elsewhere [16]. Fracture energy was measured using 4J instrumented impact hammer (CEAST, Italy) as the integral under the force-time curve.

III. RESULTS AND DISCUSSIONS

A. Effect of fiber type on the G_{IC} measured under impact loading

At a given constant vf, aspect ratio and fiber diameter, the model predicted the largest increase of GIC 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 (Figure 1). At constant fiber volume fraction, vf = 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 GIC only by 36% compared to the neat resin. Average standard deviation of less than 15% has been obtained for all the composites tested. Comparing the experimental data with the predictions based on Eq.(1), one can conclude that a reasonably good egreement exists between experimental data and the model predictions..

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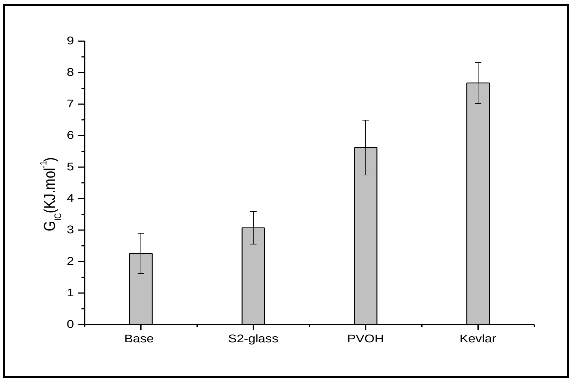

Figure 1. The G_{ICC} determined from the instrumented Charpy impact test data for the denture base resin modified with S2-glass,
PVOH and Kevlar 29 fibers, respectively. The v_f = 0.10, initial L/d = 267 and the tests were performed at the impact speed of 1 m/s, at 23 °C and RH = 80%.

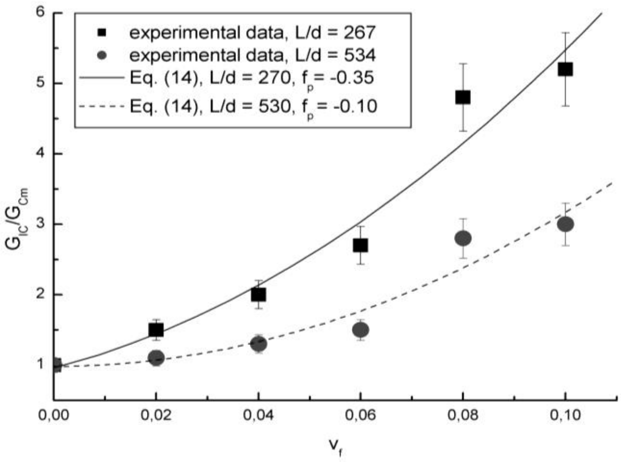

Figure 2 Comparison of the relative GICC/GICM for the denture base resin modified with PVOH fibers of 2 aspect ratios (initial L/d=267 and L/d=534) calculated using equation (14) with experimental data. Fiber orientation factor, fp, has been determined from the polished SEM photographs of the fracture surfaces using the image analysis software (HarFA). The fp= - 0.35 was determined for L/d=267, and the fp = - 0.1 was determined for L/d=534.

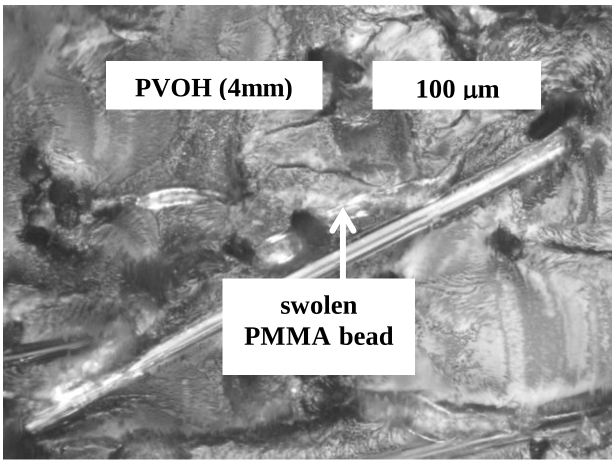

Figure 3 Morphology of the short PVOH (b) modified denture base resin as revealed by the laser scanning confocal microscopy. The "dough" technology used to process the mixture of PMMA beads with the MMA monomer liquid produces heterogeneous morphology consisting of swollen beads embedded in a PMMA continuum. These swolen beads possess different properties and cause stress concentration in the matrix resulting in multiple crack initiations.

IV. 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, GIC, of the composite containing small fiber volume fraction. Even though the fiber pull-out and de-bonding are the main contributions to the GIC, 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 4mm fibers resulted in significantly greater enhancement of composite fracture toughness compared to the 8mm fibers, at the same vf. 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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AUTHORS PROFILE

Dr. E.N .Ganesh, having 24 years of Teaching Experience out of which 8 years as Principal and 3 years as Dean at present. I am a M.Tech Graduate from **IIT Madras** in Microelectronics and VLSI Design, and PhD from JNTU Hyderabad in **Nanotechnology with Gold Medal**. I have Executed 6 Funded Projects with 5 patents granted. 5 More Patents are Published and all of them are in final evaluation stage. DST STTP Sponsored Funded one Crore project is in progress and at final stage to be submitted. I am Guiding 13 PhD students, 5 from Anna University and all of them awarded degree. I am having 48 Scopus indexed International Journals and 15 SCI Journals (Annexure I) with total 160 Publications to my credits. I have executed 12 Consultancy Projects for the cost of 60 Lakhs. I have more than 60 International Conference Publications. Twenty best Conference paper awards with Distinguish Faculty, Researcher and Excellence in teaching awards, reviewer and editor for 8 International Journals with four books Published (one International Publication).

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