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A comparison of mechanical properties of discontinuous Kevlar 29 fibre reinforced bone and dental cements

B. POURDEYHIMI
Department of Textiles and Consumer Economics, University of Maryland, College Park, Maryland 20742, USA

H. D. WAGNER
Department of Materials Research, Weizmann Institute of Science, Rehovot 76100, Israel

P. SCHWARTZ
Department of Textiles and Apparel, Cornell University, Ithaca, New York 14853–4401, USA

A comparative study of the fracture behaviour of Kevlar 29 reinforced bone and dental cements is undertaken using both linear elastic and non-linear elastic fracture mechanics approaches. Results from both approaches reflect improved fracture toughness at very low fibre contents. Flexural modulus is not apparently improved in either system, and flexural strength is only improved in the bone cement system probably because of poor interfacial bonding and the presence of voids in the dental cement. In all cases, however, bone cement is seen to be superior to dental cement. This is interpreted in terms of smaller voids and better fibre distribution due to the lower viscosity of the bone cement material. When compared to carbon–polymethyl methacrylate (PMMA) cements, Kevlar 29 reinforced systems appear to be superior. More work is underway to optimize the properties of these systems with regard to structural parameters.

1. Introduction

The pioneering work of Charnley [1, 2] in the late 1950s has resulted in the routine use of self-curing acrylic bone cements for partial and total joint replacements. The material was first used for orthopaedic implant fixation in total hip prosthesis and has since been used for stabilization of hip, knee, shoulder, elbow and other prostheses. Prior to their introduction into orthopaedic surgery, acrylics had been used as dental materials for some time with much success [3].

Another important application of acrylic bone cement is in its use with conventional fixation plates for stabilizing pathological fractures at sites of malignant neoplastic bone [4]. Bone cement is also used for building up a region of the bone which may have been lost by disease [5].

Acrylic bone cement consists essentially of polymerized polymethyl methacrylate (PMMA) powder and methylmethacrylate (MMA) liquid monomer. Initiators such as benzoyl peroxide are present in the powder. In addition, a variety of additives may also be present. For example, it is common to find low levels of barium sulphate ($BaSO_4$) which is intended to provide radiopacity to the cured cement. This makes postoperative X-ray follow-ups easier. Dental cements essentially contain the same materials but may also contain small amounts of colourant. The amount and types of additives may vary depending on the type of the commercially available cements.

When used for orthopaedic implant fixation, the bone cement serves as an intermediate phase between the high modulus metallic implant and lower modulus bone, and its role is one of a mechanical interlock. "By contouring to the implant and bone profiles, the cement serves to eliminate high-contact stress points at the bone interface". Such high contact stresses were responsible for the failures of earlier implants. Pressure necrosis of the bone caused by devascularization at the contact points led to bone resorption and implant instability [5].

Despite a relatively good success rate, major problems still remain. Under operating room conditions, where the PMMA powder and MMA liquid monomer are mixed and hand applied or injected under relatively low setting pressures using a special large syringe, air entrapment results in a very imperfectly structured acrylic bone cement. The cement is full of voids and imperfection folds [5–6] which act as stress raisers giving rise to a loss in mechanical properties.

A second, and more serious, problem encountered is the implant loosening at the cement–bone or cement–prosthesis interfaces. This may arise from the poor adhesive properties of the cement [7], or poor cement packing [7, 8], resulting in loss of mechanical properties aggravating even further the mismatch in mechanical properties of the bone–cement–implant structure. Indeed, it has been stated that the Young's moduli of the structure are in ratios of about 20 : 2 : 200 GPa [8]. Other possible causes of poor

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TABLE I

Fibre type and properties	Fibre used (%)	Tensile strength (MPa)	Compressive strength (MPa)	Flexural modulus (MPa)	Tensile modulus (GPa)	Compressive modulus (GPa)	Flexural modulus (GPa)	Fracture toughness (MPa $m^{1/2}$)
Unreinforced Cements*		24 [14]	68–103 [25]	48–52 [13]	1.95–2.29 [29]	1.7–2.14 [25]	2.0 [30]	0.88–1.03 [27]
		28.9–32.6 [19] 30.8 [16] 21.6–33.9 [29]	82–85 [13] 84.4–89.2 [26]	49.9–60.2 [29] 51.2–68.4 [25]	2.00–3.05 [5] 2.76 [14]		2.1–2.3 [13]	1.18–1.21 [29] 1.23–1.51 [26] 1.32–1.46 [28] 1.53 [16]
Carbon [5, 14] L = 6 mm	2.0 (vol)	38	–	–	5.52	–	–	–
Graphite [13]	1.0 (wt)	–	–	48	–	–	2.3	–
L = 6 mm	2.0 (wt	–	–	51	–	–	2.5	–
	3.0 (wt)	–	–	48	–	–	4.1	–
	10.0 (wt)	–	12–23	43–51	–	–	4.4–4.9	–
Graphite [26] L = 1.5 mm	2.0 (vol)	–	88.2–88.8	–	–	–	–	1.61–1.88
Kevlar 29 [16]	1.0 (wt)	36.1	–	–	–	–	–	1.88
L = 13 mm	4.0 (wt)	38.2	–	–	–	–	–	2.31
	7.0 (wt)	42.8	–	–	–	–	–	2.85
Kevlar 29 [17]	2.0 (wt)	–	79.4	–	–	1.88	–	–
L = 13 mm	4.0 (wt)	–	85.2	–	–	1.56	–	–
Kevlar 29 [18]	1.0 (wt)	–	–	70.9	–	–	1.11	1.06
L = 3.2 mm	7.0 (wt)	–	–	52.9	–	–	0.87	1.96

*Data for surgical Simplex, Simplex P, Zimmer and PMMA (Kerr–Sybron Corporation) have been gathered.

mechanical performance may be chemical and thermal necrosis, shrinkage of the cement as well as biological factors. Of these, thermal and chemical necorsis are related to cement properties. The curing temperature reaching as high as 83° C has been blamed as the cause of thermal bone necrosis, while others claim that the necrosis may be brought about by the release of MMA monomer which may also find its way into the blood [9, 10].

Whatever the cause, it is clear that such microstructural changes result in inferior mechanical properties. Indeed, at lower joint extremities where the stresses are of high magnitude, static and dynamic stressing may result in cement break-up, part loosening or both [11].

Two current trends in research have endeavoured to improve the performance and, therefore, the lifetime of surgical cements: (i) by developing porous cements so as to allow bone ingrowth [9], and (ii) through incorporation of high performance fibres, such as carbon and Kevlar [poly(*p*-phenylene terephthalamide)]. The results found in the literature [12–19, 25–30] are summarized in Table I. The use of fibres has resulted in improved mechanical properties and has the added advantage of slowing the rate of polymerization, thus decreasing the exotherm [13]. These studies, however, have failed to provide answers to such key questions as:

1. What is the optimum fibre length and content in respect to mixing and mechanical performance?
2. What is the optimum fibre–matrix bond strength and how to control the bond strength?
3. What is the average lifetime of the cements under simulated life testing?
4. What is the optimum viscosity to minimize void content?

Indeed, it is surprising that none of the reports published have paid any attention to optimization of the fibre reinforced systems.

In this work, a failure analysis of Kevlar 29–PMMA dental and bone cements is undertaken. The data for the reinforced dental cements were generated in our laboratories in a recent study [18], and we use these as a baseline, for comparison purposes, with bone cement in the present work.

2. Experimental prodedure and method

The materials used were low viscosity bone cement (Zimmer, Warsaw, Indiana) and dental cement (DUZ A11). The powder and the liquid were mixed at room temperature using the customary 2:1 powder:liquid ratio. Apart from the control samples, reinforced cements were fabricated using 3.2 mm long Kevlar 29 fibres (E. I. du Pont de Nemours & Co). The fibre was mixed with PMMA at 1 and 7 wt % prior to mixing with MMA. The cements were mixed well using a thin stainless steel blade and moulded immediately in a mould of two glass plates separated by 5 mm thick rubber spacers. The mould was placed in a circulating air oven at 37° C and allowed to cure for at least 24 h before any tests were performed. The dimensions of the plates were 25 × 25 × 0.6 cm. Three-point bend

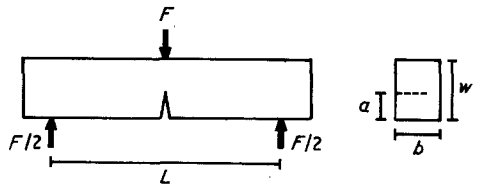

Figure 1 Three point bend testing configuration.

test beams (0.6 × 0.6 × 3 cm) were then machined from the plates and sharp notches of various depths were inserted using a saw-tooth edge solid carbide circular blade (Technology Associates Inc., Wilmington, Delaware). The notations used for the beam dimensions are shown in Fig. 1. The specimens were tested in 3 point bend loading with a span of 2.5 cm using an Instron Model 1130 testing machine, at a displaement rate of 0.5 cm min^{-1}. The sample size was six specimens per data point in all cases.

Flexural strength and modulus were computed using the following formulae:

$$\sigma = 1.5\ FL\ bw^{-2} \quad (1)$$

and

$$E = kL^3\ 4\ bw^{-3} \quad (2)$$

where F is the maximum load and k is the slope at the origin of the load–displacement curves.

Fracture toughness was computed using the following expression for the K calibration [20].

$$K_{Ic} = \frac{1.5\ FL}{bw^{3/2}}\ Y(a/w), \quad (3)$$

where

$$Y(a/w) = 1.93(a/w)^{1/2} - 3.07(a/w)^{3/2} + 14.53(a/w)^{5/2} - 25.11(a/w)^{7/2} + 25.8(a/w)^{9/2}$$

for a span:width ratio ($L:w$) of 4.

Fracture toughness analysis was also performed using the J-integral approach [21]. J at constant displacement for a specimen thickness b, is:

$$J = \frac{1}{b}\frac{\delta U}{\delta a}\bigg|_{\text{displacement = constant}} \quad (4)$$

where U is the potential energy and a is the crack length.

J is calculated experimentally from load–displacement curves for specimens with different crack lengths. The curves are integrated to determine the work done in loading to a given displacement. Work is measured at pre-determined displacements so that a plot of normalized strain energy (U/b) against crack length can be made for each displacement. J is measured by taking the negative of the slopes of the plots of strain energy against crack length. A plot of J against displacement can subsequently be made. To determine the critical value of J for crack initiation, J_{Ic}, a critical value of displacement must be avoided. Crack initiation corresponds to the point where the load first begins to drop off. Therefore, from each of the load–displacement curves, a critical value of displacement is determined. J_{Ic} can subsequently be

determined by taking the value of J from plots of J against displacement, at that critical displacement.

The use of J-integral has been well described in the literature [21–23] and J_{Ic} in randomly oriented short fibre composites has been shown to agree well with the critical intensity factor obtained using R-curve analysis [24].

The fracture mechanics approach of using K_{Ic} to predict unstable crack extension in a cracked notched body is most useful for the limited case of linear elastic plane strain fracture. By contrast, the J-integral is a fracture criterion which also includes elastic-plastic to fully plastic behaviour. This method is believed to be more appropriate for PMMA composites whose stress–strain behaviour shows some deviation from linearity. The assumption that stress–strain behaviour of PMMA and PMMA composites are linear until failure has previously allowed the use of linear elastic fracture mechanics to compute plain strain fracture toughness. It has been shown, however, that the behaviour is perhaps non-linear elastic [4, 15, 18], the degree of non-linearity being dependent on such variables as strain rate, temperature, fibre content and void content.

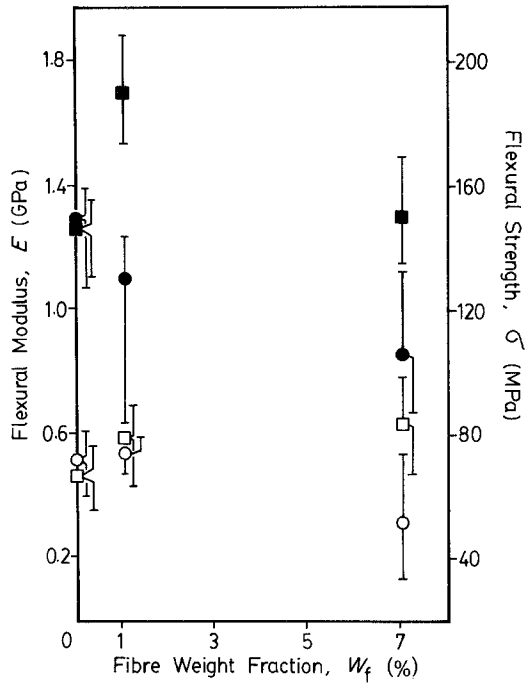

Figure 2 Flexural strength and modulus as a function of fibre content. Open symbols, strength; closed symbols, modulus. Circle, Kevlar 29–dental cement; square, Kevlar 29–bone cement.

3. Results and discussion

3.1. Flexural strength and modulus

Flexural strength and modulus are plotted as a function of fibre content in Fig. 2, and a summary of the results of the mechanical properties obtained in three-point bend loading is given in Table II.

It may be seen that some reinforcing effect is observed for the flexural strength of the Kevlar 29–bone cement system, although the strength is seen to decrease with increasing fibre content, for the Kevlar 29–dental cement system. The increase in flexural strength of the bone cement system agrees well with the findings of Wright and Trent [16] who observed increases in tensile strength of PMMA reinforced with 13 mm Kevlar 29 fibres and graphite fibres. As will be discussed in a later section of this paper, fewer imperfections are present in the bone cement system than in the dental cement system. This arises from the fact that due to the lower viscosity of bone cement mixing is easily accomplished, fewer voids are introduced, and a superior fibre distribution is achieved.

In terms of flexural modulus, it may also be seen that the results obtained in this study show a significant decrease for the dental cement system. By contrast, at 1 per cent weight fraction the modulus of the bone cement system is greatly improved but is seen to decrease with further increase in fibre content. These results are in contrast to those of graphite reinforced PMMA where the flexural modulus increased with fibre content [26]. These results may possibly be interpreted in terms of poor interfacial bonding, poor specimen quality and the presence of voids.

3.2. K_{Ic} analysis

Fracture toughness values, K_{Ic}, are plotted against crack lengths in Figs 3 to 5. For each of the four fibre weight fractions, a constant value of stress-intensity factor, K_{Ic}, is obtained. These are shown in Table II.

K_{Ic} values do increase with increasing fibre content in both systems, the bone cement system being superior to the dental cement system. The K_{Ic} values for bone cement are similar to those of 13 mm Kevlar 29 reinforced bone cement at the same fibre weight fractions. It should be noted, however, that the length of fibres used in this study was 3.2 mm, while 13 mm Kevlar 29 fibres were used in other studies. Clearly, there is much less interfacial area with shorter fibres,

TABLE II Experimental results of the mechanical properties of Kevlar 29–PMMA bone and dental cements

Fibre content (wt %)	Flexural strength (MPa) (*CV**)		Flexural modulus (GPa) (*CV*)		K_{Ic} (MPa m$^{1/2}$) (*CV*)		J_{Ic} (KJ m^{-2}) (*CV*)	
	Bone cement	Dental cement	Bone cement	Dental cement	Bone cement	Dental cement	Bone cement	Dental cement
0	67.4 (7.1%)	70.3 (13.0%)	1.28 (8.7%)	1.28 (11.2%)	1.47 (7.5%)	0.99 (4.1%)	1.56	0.654
1	74.2 (11.9%)	70.9 (6.0%)	1.70 (14.2%)	1.11 (22.4%)	2.08 (12.1%)	1.06 (8.6%)	2.28	0.860
7	82.6 (24.7%)	52.9 (32.1%)	1.30 (15.6%)	0.87 (26.3%)	2.61 (17.7%)	1.96 (5.2%)	4.81	3.002

* *CV* is the coefficient of variance.

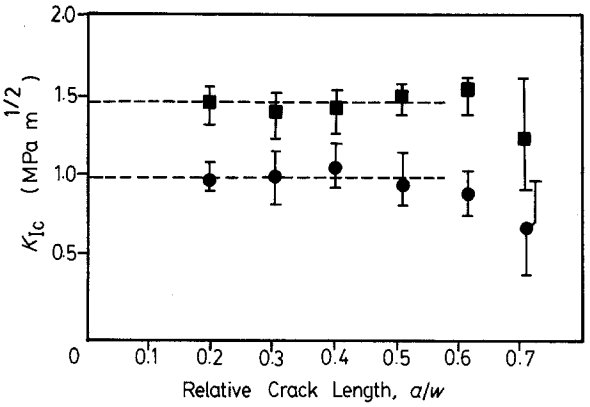

Figure 3 Fracture toughness as a function of crack length for PMMA cement specimens. ●, Kevlar 29–dental cement; ■, Kevlar 29–bone cement. $W_f = 0$.

possibly resulting in less energy being dissipated. It is probable that longer fibres in a low viscosity material such as that used in this study might show further improvements.

Compared to graphite, 1 wt % reinforcement with 3.2 mm Kevlar 29 fibres, corresponding to a volume fraction of about 0.8 %, yields slightly higher K_{Ic} values than 2 vol % reinforcement with 1.5 mm graphite fibres. This could be due to fibre type, fibre length or interfacial bonding.

These are indications of a potential trend and more work is needed to fully realise the potential of these systems.

3.3. *J*-integral analysis

Values of the *J*-integral for each of the weight fractions were calculated from load–displacement curves following the interpretation outlined earlier. The area under the load–displacement curves at several total deflections, was found using a polar planimeter. The energy at each of the displacements was plotted as a function of crack length. The slopes of these curves are equal to $(\delta U/b)/\delta a$, the change in potential energy per unit change in crack length. J is $-\delta U/\delta a$, normalized to unit thickness.

The strain energy normalized to the thickness for each material is plotted against crack length for several values of displacement and is shown in Figs 6

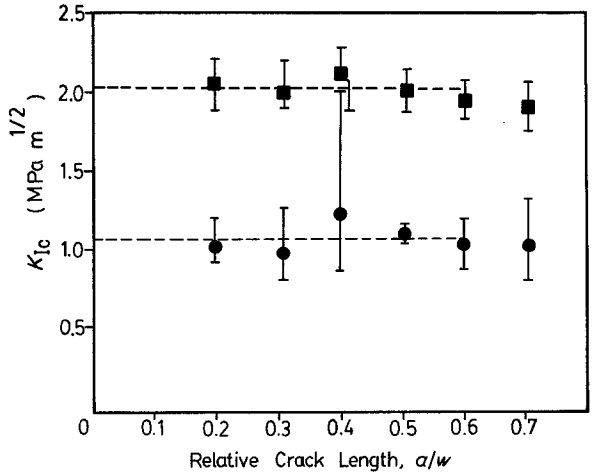

Figure 4 Fracture toughness as a function of crack length for 1% weight fraction Kevlar 29 PMMA cement specimens. ●, Kevlar 29–dental cement; ■, Kevlar 29–bone cement.

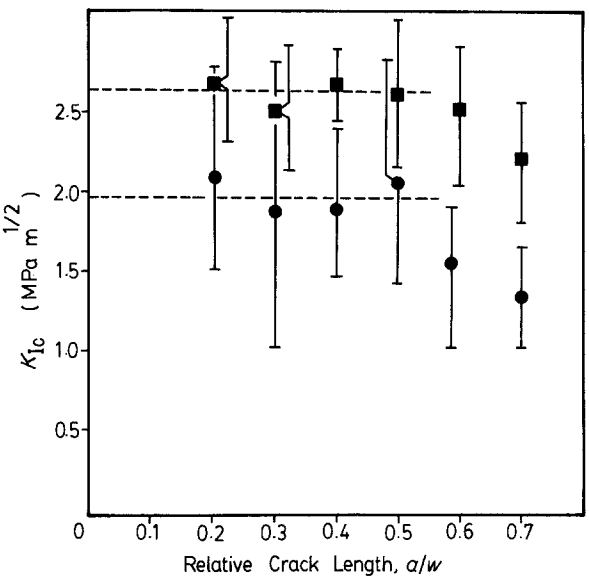

Figure 5 Fracture toughness as a function of crack length 7% weight fraction Kevlar 29 PMMA cement specimens. ●, Kevlar 29–dental cement; ■, Kevlar 29–bone cement.

and 7, the *J*-integral being the slope of the resulting curves. A linear regression was used for determining the slope of the lines. The *J*-integral (i.e. the negative of the slopes) is plotted for each of reinforced PMMA cements in Fig. 8. The *J*-integral curves, according to theory, follow a parabolic form for elastic behaviour, and a straight line for rigid plastic behaviour. The curves shown in Fig. 8 were obtained using parabolic regression. The actual behaviour of acrylic cements is probably a combination of these two extremes as observed in Fig. 8. The J_{Ic} results are reported in Table II. These values were determined at their corresponding critical displacements shown in Fig. 9.

It has been shown that J_{Ic} is related to K_{Ic}; this relationship can be stated in terms of [20].

$$J_{Ic} = \frac{(1 - \nu^2)}{E} K_{Ic}^2 \qquad (5)$$

where ν = Poisson's ratio and E = Young's modulus. Using a Poisson ratio of 0.35 for all materials, critical stress-intensity values of 1.48, 2.06, and 2.6 are obtained which agree well with the observed values of K_{Ic}.

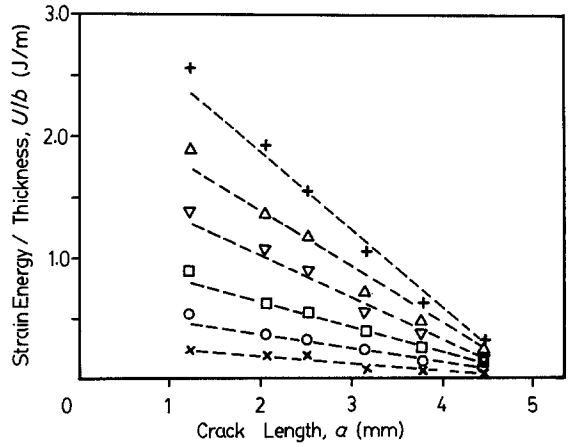

Figure 6 Strain energy per unit thickness as a function of crack length for dental cement specimens. +, 0.28; △, 0.24; ▽, 0.20: □, 0.16; ○, 0.12; ×, 0.08. $W_f = 0$.

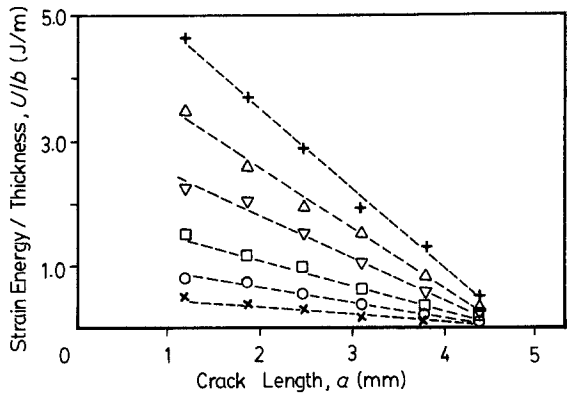

Figure 7 Strain energy per unit thickness as a function of crack length for bone cement specimens. Displacement: +, 0.28; △, 0.24; ▽, 0.20; □, 0.16; ○, 0.12; ×, 0.08. $W_f = 0$.

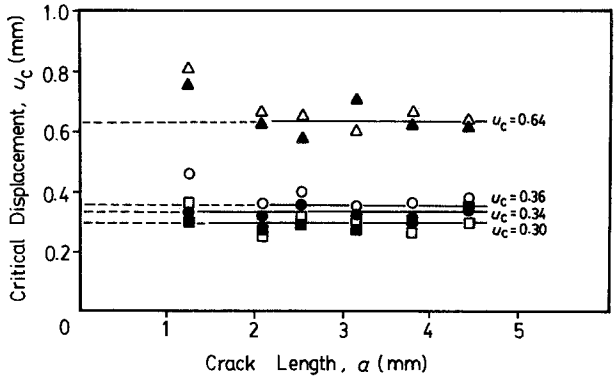

Figure 9 Critical displacement as a function of initial crack length. Closed symbols, Kevlar 29–dental cement; open symbols, Kevlar 29–bone cement. W_f = triangle, 0.07; circle, 0.01; square, 0.

3.4. Electron microscope observations

Several bone cement samples were observed by scanning electron microscopy (SEM), using a JEOL JSM 35 instrument. As in our previous study [18], a relatively large number of voids of various sizes could be observed, although the mean diameter of a typical void, about 150 to 200 μm, was somewhat smaller than in dental cements. Few thermal cracks were observed. As in dental PMMA, a clear gap existed at the matrix–fibre interface, reflecting poor interfacial bonding.

Finally, typical tensile failure (by splitting) and flexural failure (by piling up of kink bands) of Kevlar 29 were observed as before [18]. These failure modes represent high energy absorption mechanisms which are specific to Kevlar fibres.

4. Conclusions

From the results of our experiments, we conclude the following:

1. The fracture toughness of Kevlar 29 reinforced bone and dental cement increases with increasing fibre weight fraction for weight fractions ranging to 7%. Our shorter (3.2 mm) fibres yielded consistent results compared with earlier studies [16] using longer fibres (13 mm). This increased toughness we attribute to the partially bonded interface which forces an advancing crack to deflect along the length of the fibre, expending considerable energy as the bonding between fibre and interface is broken down. We have argued elsewhere [18] that this increase in fracture toughness is maximized by imperfect bonding, dropping off as the bonding either moves to perfect, on one hand, and none on the other.

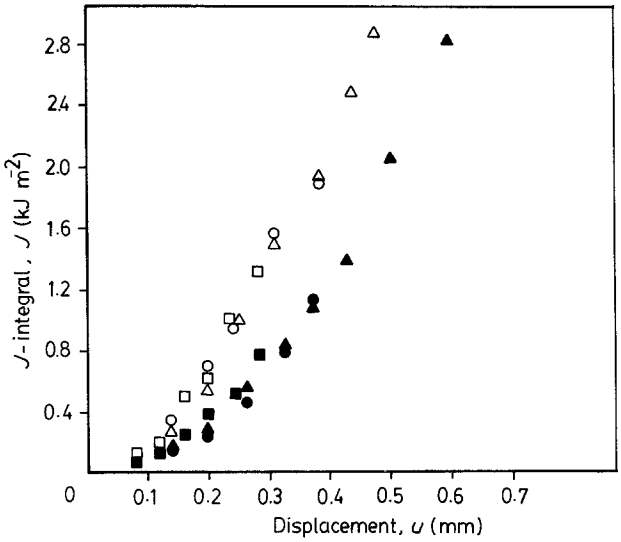

Figure 8 J-integral as a function of displacement for PMMA cement specimens. Closed symbols, Kevlar 29–dental cement; open symbols, Kevlar 29–bone cement. W_f = triangle, 0.07; circle, 0.01; square, 0.

2. Both dental and bone cement systems show an initial, small strength increase as the Kevlar 29 fibres are incorporated with the dental cement system losing strength with higher fibre concentration while the bone cement shows relative insensitivity. This is most probably due to the more thorough fibre mixing and reduction of voids in the bone cement. This might also explain the brief rise in modulus of the bone cement while the dental cement experiences a steady decline.

3. For all weight fractions the fracture toughness is relatively insensitive to initial crack length with the drop-off occurring in the 0.6 to 0.7 area. Because this effect is seen in $W_f = 0$, we speculate that the phenomenon is intrinsic in the PMMA, and not affected by the fibres, at least not for the range of W_f used in our studies.

4. We conclude, as has been argued previously [24] that the *J*-integral technique provides an easy, accurate method for predicting the fracture toughness of short-fibre composites, in our case PPTA–PMMA composite materials.

In our studies, we have seen and understood the necessity for good mixing to take place between fibres and matrix. We are currently developing better mixing techniques which, we feel, will allow us to obtain higher strength and fracture toughness combined with a more consistent modulus. We plan to report these results in the near future.

Acknowledgements

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