Analysis of push-out test data based on interfacial fracture energy

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Push-out testing is frequently used to assess the interfacial shear strength developed at a bone–biomaterial interface during *in vivo* experiments. The aim of the present research was to assess the *in vivo* performance of a novel substrate/coating combination and to introduce a more rigorous fracture mechanics analysis of the push-out test data. An adhesively bonded hydroxyapatite (HA), and a Ti-6Al-4V alloy plasma sprayed with HA, were implanted in female New Zealand white rabbits for up to 6 months in duration. After death, push-out tests were carried out and the shear strength was calculated in the conventional way, together with microscopical examination of crack paths. A finite element model was drawn up representing four potential failure mechanisms. The measured ''failure shear strengths'' in conventional analysis were approximately equal for the two coatings. However, J_C at failure calculated from the model was 210 J m^{-2} at the novel adhesively bonded HA/bone interface and 5 J m^{-2} at a conventional titanium/plasma-sprayed HA interface. The conventional shear strength approach is strongly test dependent, and we believe that the fracture energy approach represents a more rigorous analysis of the real failure criterion in the implant/host tissue structure.

1. Introduction

The strength of the interface between a biomaterial and bone is critical to the long-term performance of any load-bearing implant. The effects of failure or damage at this interface in conventional implants are well understood. Despite recent improvements in the stability of this interface with the advent of hydroxyapatite coatings, its mechanical integrity remains an important consideration, especially when dealing with new coatings for which the bonding characteristics of the bone/biomaterial interface are unknown.

Push-out testing is often utilized as a method of assessing the mechanical strength of a bone–biomaterial interface developed during *in vivo* experiments, generally concentrating on the bone-bonding performance of HA-coated Ti-6Al-4V [1, 2] or the development of implant–bone interface strength with time [3–5]. The test measures the interfacial shear strength developed between a biomaterial and bone.

Previously, a finite element analysis has been performed on the push-out test configuration [6] in which the effects of frequently varying parameters on the interfacial shear strength were calculated. The model was based on implantation of a cylinder of material in cortical bone. It was discovered that the interfacial stress distribution was strongly dependent upon implant–support jig distance and on the stiffness of the implant itself. Also a dependence of interfacial stress level on cortical bone thickness was reported, although the actual stress distribution was unaffected. The modeling led to the suggestion that interfacial stress could be kept fairly constant over the fracture surface only if the Young's modulus of the implant was less than ~ 50 GPa and the implant–support jig distance was greater than 0.7 mm. Thus, a (standardized) test may be used to study trends during follow-up periods but comparison of different tests, and especially comparison between different implant materials is severely flawed.

The aim of the present research was to assess the *in vivo* performance of a novel substrate/coating combination and to introduce a more rigorous fracture mechanics analysis of the push-out test data.

2. Experimental procedures

Two substrate/coating combinations were studied:

- Ti-6Al-4V with a plasma-sprayed HA coating (Plas-HA). Plasma spraying was carried out by Plasma Biotal. An HA coating of thickness 50 μm was deposited.
- Ciba Geigy 5052 epoxy (CG5052) with an

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adhesively bonded hydroxyapatite coating (Ad-HA) which has the advantage of providing a more even stress distribution across a bone-implant interface [7], improved mechanical properties [8, 9] and good biocompatibility [10]. The Ad-HA coating used a substrate of the above epoxy, reinforced with 40% by weight of fine HA particles (approximately 1 μm in diameter), coated with a layer of coarse ($\sim$ 250 μm diameter) HA particles, applied while the previous layer was still uncured to obtain good adhesion. These particles were intended to provide the biocompatibility and thus constituted the outer coating. The Ad-HA was allowed to cure at room temperature for 24 h. A post-cure of 1 h at 100 °C was then carried out.

The subsequent *in vivo* implantation and mechanical testing were carried out at the Royal Free Hospital School of Medicine, Department of Histopathology using female New Zealand white rabbits as the animal model. Under general anaesthesia, a 3.5 mm diameter hole was drilled in the femoral condyle. Implants (3.5 mm diameter) of the biomaterials listed above were inserted into this hole by press-fitting. These implants were then left *in situ* for periods of up to 6 months. After sacrificing the animals, histological sections were used to study bone formation adjacent to the implant, and mechanical testing was undertaken to evaluate the strength of the bone–biomaterial interface, using the push-out test. To obtain the specimens for push-out testing, femurs were retrieved and sectioned immediately following death of the rabbits, ensuring that the long axis of the specimen was parallel to the axis of the implant. A representation of the push-out test set-up including test and specimen geometries is shown in Fig. 1. The push-out tests were carried out at a cross-head speed of 1 mm min^{-1}, and conventionally, a mean value of the shear stress was calculated.

3. Results and discussion

Measurements of interfacial shear strengths for the different materials after 0 and 6 weeks implantation are given in Table I. The strength of the interface is clearly developed over the first 6 weeks following implantation, during which bone–biomaterial bonding takes place. It can be seen that the HA particle coating has a significant effect on the interfacial shear strength developed by the Ad-HA implants (an improvement of $\sim$ 50% compared with uncoated material). However, using conventional analysis based on interfacial shear strength measurements, it can be seen that there is no difference between plasma-sprayed HA on a titanium substrate and adhesively bonded HA on an epoxy substrate.

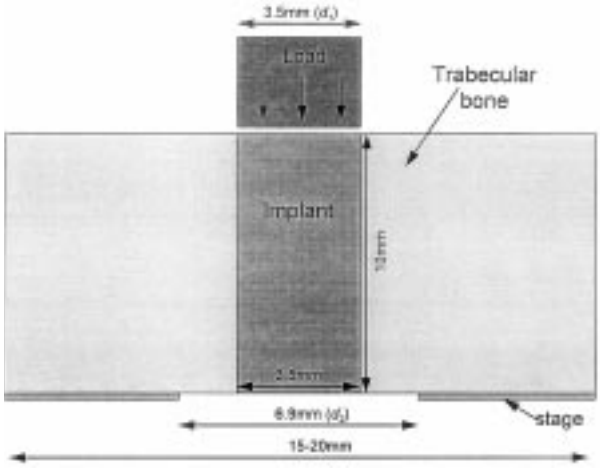

Figure 1 Cross-section of the push-out test, showing the specimen, pin and stage with appropriate dimensions.

Microscopic examination of the samples after push-out revealed that the Plas-HA coatings were generally adhered to the bone and not to the titanium implant (Fig. 2), demonstrating failure at the coating/substrate interface. In contrast, the Ad-HA coatings are characterized by failure at the coating/bone interface, with the coating remaining well bonded to the substrate (Fig. 3).

Although the actual failure strengths of the Ad-HA and Plas-HA were almost identical, there was a distinct difference in the failure path. This could be due to better bonding between the plasma-sprayed HA and bone, but this was not borne out by results [11] for bone growth rate and integration to the two materials, which showed no significant difference between the alternative coatings. A more likely explanation is that the interfacial shear stress does not fully describe the processes taking place at the interface when the specimens are loaded.

The measurement and interpretation of shear failure stresses in this conventional manner as described above has three principal drawbacks: (i) the variable parameters (e.g. d_1 and d_2 (Fig. 1)) of the push-out test will significantly affect the failure loads required and the stress distribution across the interfacial region; (ii) the calculated ''interfacial shear stress'' assumes that the load is evenly distributed over the entire fracture surface; (iii) the failure process will involve cracks nucleating at imperfections in the material and at stress concentrations. As a consequence of these limitations, further investigation of the failure process was undertaken.

A more rigorous analysis of the push-out test data may be introduced by incorporating fracture mechanics principles into a finite element model. During any fracture process, the strain energy release rate G_C is a material property independent of test geometry and can be applied to coating/substrate interfaces [7, 9], thus allowing a fracture mechanics-based prediction of failure for the different coated materials.

The finite element model was drawn up using the package FRANC2D/L, a finite element analysis (FEA) package specifically designed for fracture mechanics. The loading and boundary conditions are summarized in Fig. 4, with the central axis being fixed in the x-direction but allowed to move in the y-direction and the area restrained by the jig was fixed in the y-direction only. The failure load was applied as an evenly distributed load over the implant. To simulate the experimental materials, the finite element model incorporated (i) a 50 μm layer of plasma sprayed HA on a titanium alloy substrate, bonded to trabecular bone, and (ii) a 100 μm layer of Ad-HA on an epoxy substrate, again bonded to trabecular bone. The material properties used in the analyses are shown in Table II. All of the materials are assumed to be linear

TABLE I Interfacial shear strengths measured in the push-out tests

Material	σ_{mean} (MPa)	Standard error (MPa)	No. of samples tested
CG5052 (uncoated, 0 w)	0.01	0.00	6
CG5052 (uncoated, 6 w)	2.40	0.82	5
CG5052 (HAP-coated, Ow)	0.28	0.05	6
CG5052 (HAP-coated, 6 w)	3.54	0.50	5
Ti (HAP-coated, 6 w)	3.52	0.67	6

elastic and isotropic for the purpose of the model with constant elastic properties being assumed throughout.

The model was set up to measure the mode I and II contributions to the stress intensity factor using the *J*-integral method, thus allowing comparison with strain energy release rates measured experimentally for the coating/substrate interface. Under linear elastic conditions the elastic–plastic energy release rate, *J*, is equal to *G*, and thus if the modeled crack path represents the path preferred by an actual crack

$$J_I + J_{II} = G_C$$

where J_I and J_{II} are the mode I and II components of *J*, respectively. Thus, if the material's critical strain energy release rate is known, it is possible to predict whether failure should occur in that material. This can be equally applied to interfaces between dissimilar materials.

Further assumptions in this model include: (i) interfaces are well bonded, without regions of discontinuity which may act as stress raisers; (ii) the jig is undeformable and completely fixed; (iii) there is only a single crack initiating at the material's surface; and (iv) crack propagation, as opposed to initiation, is assumed to be the factor controlling the rate of crack growth.

The FE model highlighted areas of high stress concentration in the trabecular bone near the implant/bone interface where the load was applied, and at the edge of the fixed stage holding the specimen in the trabecular bone (Fig. 5), and was applied to a number of potential failure scenarios with a crack initiating close to the stress concentration at the loading point.

The potential crack paths are annotated in Fig. 6, initiation of these cracks being assumed to be from the top surface and parallel to the bone/coating interface; the plasma-sprayed coating was modeled only for failure at the coating/substrate interface. Cracks were initially inserted 0.50 mm into the material, and calculations were performed for the next 0.25 mm crack growth in the

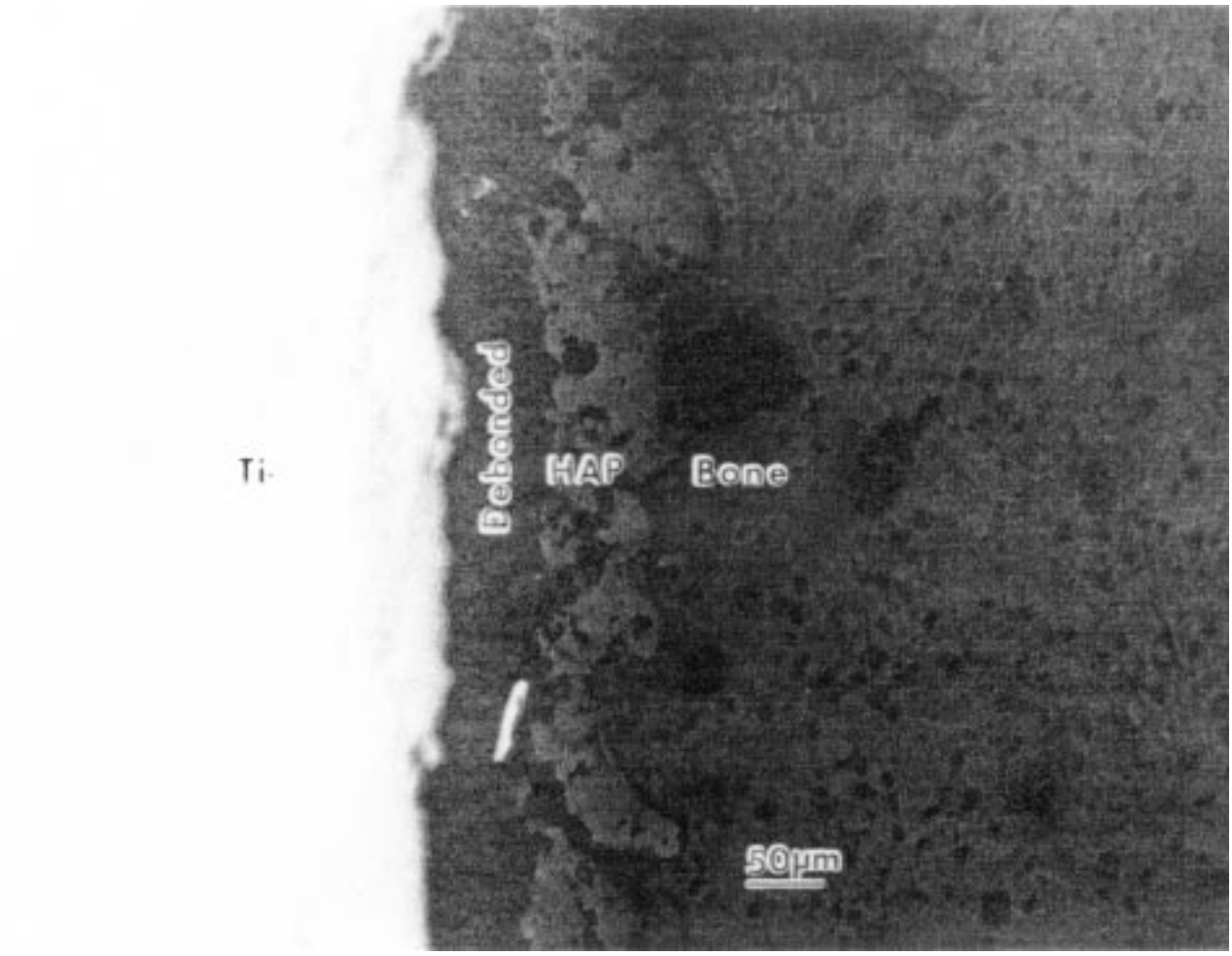

Figure 2 Optical section through the titanium–HA bone interface for a plasma-sprayed coating showing debonding of the coating from the titanium substrate.

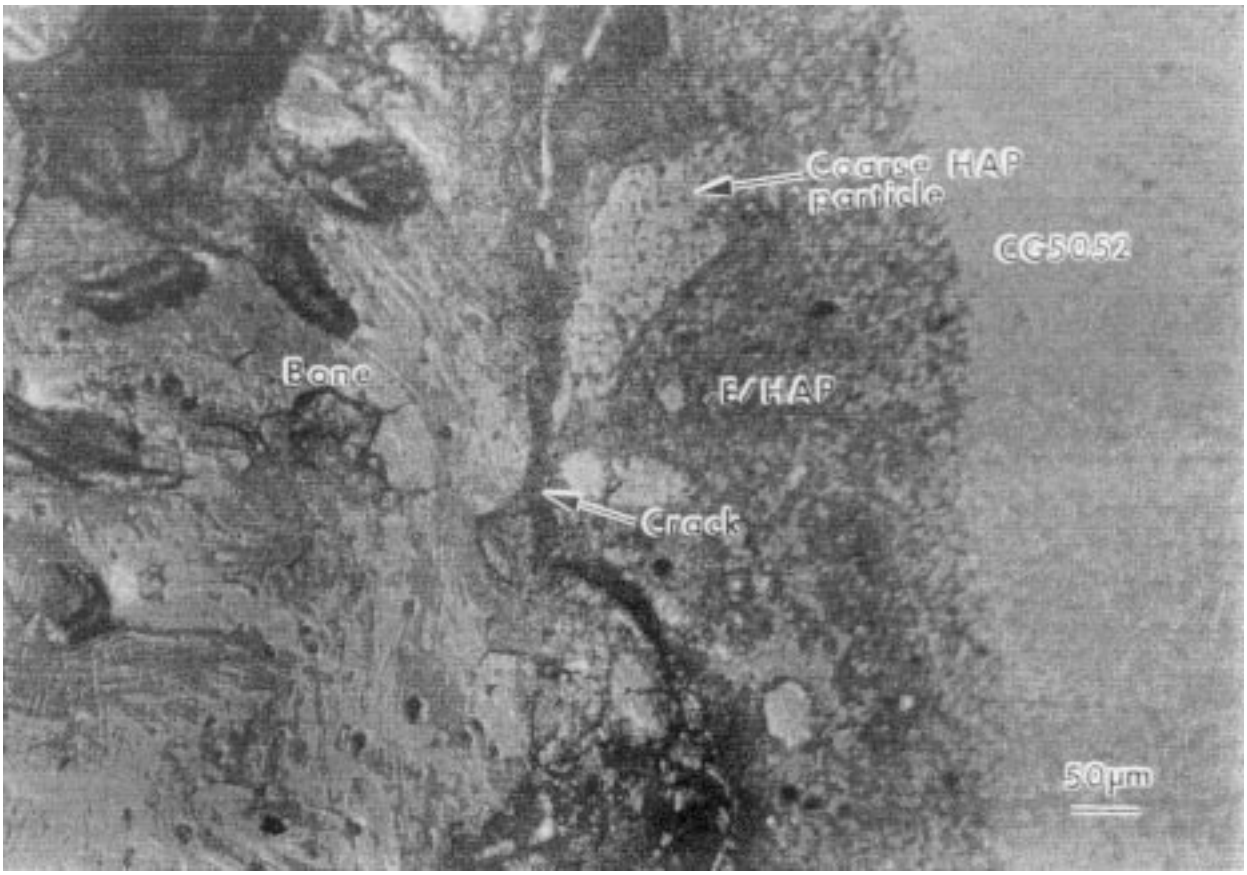

Figure 3 Section through the adhesively-bonded HA coating/bone interface clearly showing a crack between coating and bone.

TABLE II Material properties used in the finite element model

Material	Young's modulus (GPa)	Poisson's ratio
Titanium	107	0.3
CG5052	3.5	0.3
Plasma-sprayed HAP	117	0.3
HAP-filled epoxy	0.9	0.3
Trabecular bone[a]	0.5	0.3

[a]Trabecular bone was assumed to be isotropic.

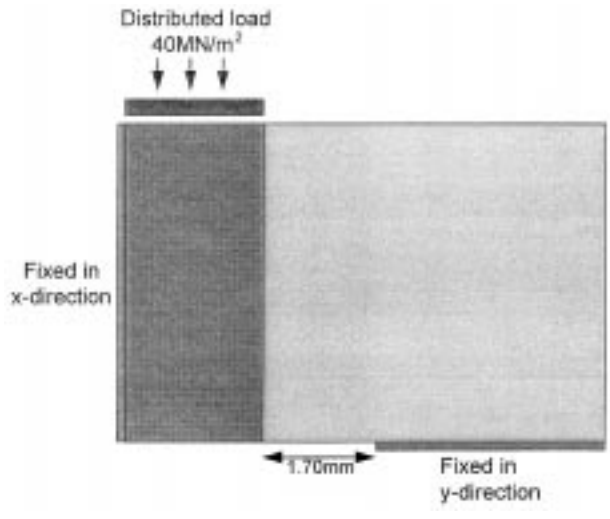

Figure 4 Loads and boundary conditions used for the finite element model.

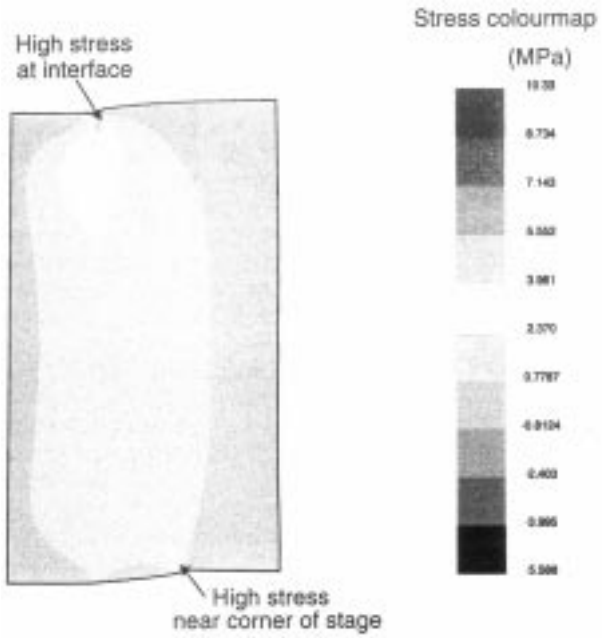

Figure 5 Stress distribution in the loaded push-out specimen.

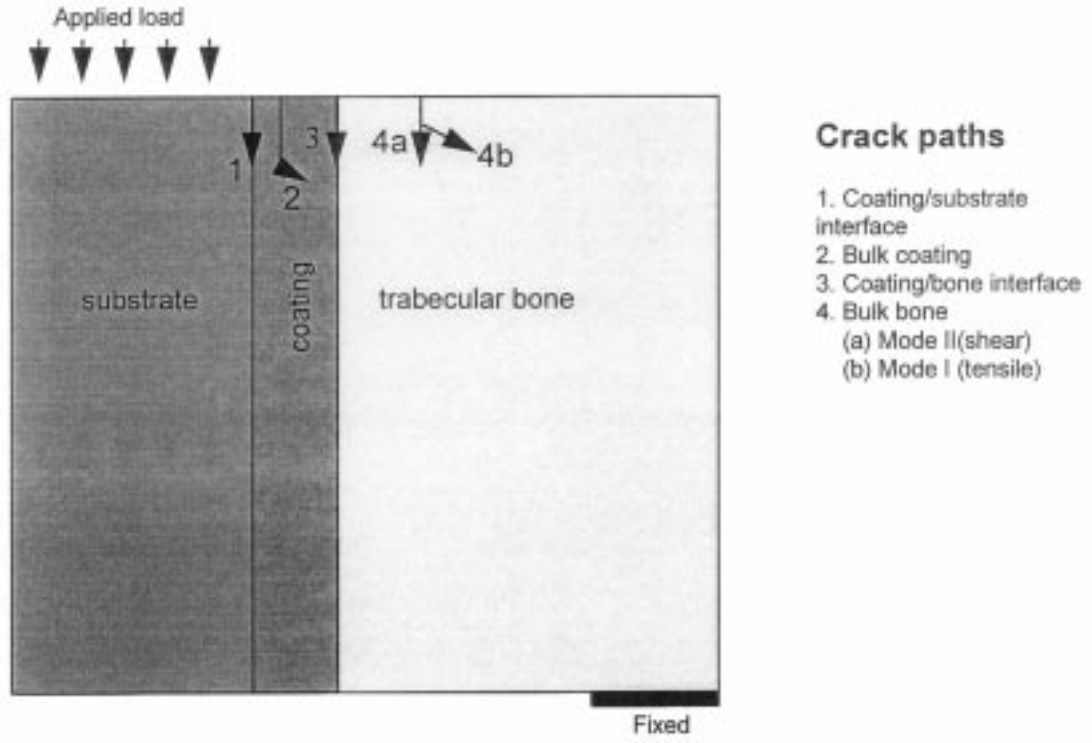

Figure 6 Crack paths considered in the model. Note that all the crack paths listed were modeled for the adhesively-bonded coating, but only path I (coating/substrate interface) for the plasma-sprayed coating.

TABLE III Comparison of calculated J-integrals against known G_C

Substrate/coating	Crack path (No.)	J_I ($J\,m^{-2}$)	J_{II} ($J\,m^{-2}$)	J_{total} ($J\,m^{-2}$)	G_C ($J\,m^{-2}$)
Ad-HA	Substrate/coating interface (1)	200	160	360	> 1000
Ad-HA	Bulk coating (2)	20	60	80	200
Ad-HA	Coating/bone interface (3)	110	100	210	–
Ad-HA	Bulk bone, mode II (4a)	40	30	70	(400)
Ad-HA	Bulk bone, mode I (4b)	2	13	15	(30)
Plas-HA	Substrate/coating interface (1)	4.8	0.2	5	< 10

The G_C values in brackets are estimates because toughness measurements for trabecular bone are not available. The values given are based on one-tenth of the measurements for cortical bone [12] which should represent a lower bound for trabecular bone based on other material properties [13], although due to the large variability with anatomical position and orientation this figure is unlikely to be accurate, and is only used as a guide.

direction indicated in Fig. 6. The elastic-plastic energy release rates, J_I and J_{II}, were compared against known critical strain energy release rates for the materials and interfaces modeled in Table III.

By comparing the values of J with G_C for the adhesively-bonded coating, it can be seen that the coating/bone interface is clearly the most likely fracture site, due to the high value of J, and the knowledge that the interface will have imperfect bonding and hence a lower critical strain energy release rate than that of bulk bone. This prediction is in accord with the experimental results. G_C was greater than J for a crack in any of the other locations, leading to non-propagation at this applied load.

In contrast, for the plasma-sprayed HA coating on titanium, the model predicted $J \sim 5\,J\,m^{-2}$ for failure at the titanium/HA interface, and this matches closely the experimentally measured value [7].

The predictions of the model are thus consistent with experimental fracture path analysis for the materials and loading configuration considered. Furthermore, the strain energy release rates at failure calculated from the model varied greatly for the different coating/substrate combinations ($\sim 210\,J\,m^{-2}$ at the adhesively-bonded HA/coating interface and $5\,J\,m^{-2}$ at the titanium/plasma-sprayed HA interface) in marked contrast to the conventional failure stress data. The fracture mechanics analysis is based on the localized stress concentration at the crack tip, and incorporates geometric and materials data; it has demonstrated the substantial difference in mechanical performance of the two coatings investigated.

In future, this computational model and fracture mechanics approach could be developed to predict potential failure sites for a load-bearing implant under *in vivo* loading conditions.

4. Conclusions

An adhesively-bonded HA coating offers a significant improvement over currently used biocompatible coatings in terms of interfacial mechanical performance. Experimental data from push-out tests has been successfully analyzed using a fracture mechanics

approach. The failure mechanisms predicted from a finite element model closely match experimental observations.

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