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# The measurement of the critical stress intensity factor ( $K_{IC}$) of pharmaceutical powders using three point single edge notched beam (SENB) testing

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*Key words*: Critical stress intensity factor; Fracture; Three-point single edge notched beam; Excipient; Drug; Brittleness index

**Summary**

The critical stress intensity factor, $K_{IC}$, of a number of pharmaceutical materials has been determined using a three-point single edge notched beam (SENB) technique. The technique was initially validated using microcrystalline cellulose and the value of the critical stress intensity factor at zero porosity, $K_{IC_0}$ of microcrystalline cellulose was found to compare reasonably well to literature data determined by four-point SENB testing. The technique utilising sawn notches was tested on a number of other important pharmaceutical materials ranging from excipients to drugs including aspirin, ibuprofen and paracetamol, It was found in general that organic pharmaceutical materials are primarily brittle or semi-brittle in their mechanical response to stress based on a brittleness index, defined as the ratio of indentation hardness to the critical stress intensity factor.

## Introduction

The fracture properties of materials are important in our understanding of how they behave during compaction and comminution. Roberts et al. (1989a) used an approach based on equation of Kendall (1978a) which describes the balance between fracture properties and ductility, i.e., critical stress intensity factor, $K_{IC}$, and yield stress respectively and were able to show reasonable agreement between the measured deformation stress and the calculated deformation stress using independently measured $K_{IC}$ and yield stress measurements for different particle sizes of sodium chloride. Kendall (1978b) had previously demonstrated that the theory of brittle/ductile transitions could account for the particle size obtained during milling. These two papers emphasise the importance of the measurement of critical stress intensity factor and its profound effect on pharmaceutical processing.

The critical stress intensity factor, $K_{IC}$, describes the state of stress around an unstable crack or flaw in a material and is an indication of the stress required to produce catastrophic propagation of a crack. It is thus a measure of the resistance of a material to cracking via tensile stresses acting normal to the crack walls.

Several techniques have been used to measure the critical stress intensity factor of powders the

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most commonly used technique is the single-edge notched beam (SENB) test in which a pre-notched rectangular beam of small thickness and width in comparison to its length is subjected to transverse loads and the load at fracture measured. To data only two researchers have used the four-point loading configuration for pharmaceutical materials (Mashadi and Newton, 1987a,b, York et al., 1990. In the former paper a single grade of microcrystalline cellulose was examined whereas in the latter paper the critical stress intensity factors of a number of grades from different manufacturers of microcrystalline cellulose were determined.

With the same material reported in the earlier study, Mashadi and Newton (1988) described a double torsion (DT) technique for the experimental determination of $K_{IC}$. They commented on the differences in extrapolated value of the critical stress intensity factor to zero porosity, $K_{ICo}$, between the two techniques suggesting that for the DT specimen higher values were due to the addition of a shearing mode of fracture. It is interesting that they suggested it is essential for the method of determination be stated when quoting values of $K_{ICo}$.

Roberts and Rowe (1989) reported on a novel method of determining the critical stress intensity factor of microcrystalline cellulose (Avicel PH101) by the use of radially edge-cracked tablets (RECT) both by edge opening, i.e., RECT (EO) and diametral compression, i.e., RECT (C). They commented on the importance of the initial crack length on the value of the $K_{IC}$ assessed using a diametral compression test, concluding that an increase in initial crack length resulted in an increase of $K_{ICo}$, attributable to the influence of plastic deformation at the loading points. In addition, the differences in $K_{ICo}$ for Avicel (PH101) between various measuring techniques were attributed to differences in crack sharpness, i.e. the RECT and DT specimens have the sharpest cracks and hence the highest values while the four point SENB test specimens have the bluntest crack and hence the lowest values.

Duncan-Hewitt and Weatherly (1989a,b) reported a technique utilising the cracking around Vickers indentations on single crystals of sucrose, adipic acid, paracetamol and sodium chloride. This method is of considerable interest and a full consideration of the technique compared to measurements on compacts will be the subject of a future paper.

All the techniques described above with the possible exception of the tablet tests, require large specimens and therefore a reasonable amount of material. This is unsuitable for the determination of the critical stress intensity factor of drugs in early stages of formulation design since in many instances the total quantity of material available is only 5–10 g. Therefore, a three point loading method requiring only small quantities of material (approximately 500 mg) has been developed using the same rig as used to measure the Young's modulus of materials (Roberts et al., 1989b, 1991).

## Theoretical

For the geometry shown in Fig. 1 the stress intensity factor of a specimen, $K_{IC}$, is calculated using Eqn 1 (Brown and Srawley, 1966):

$$K_{IC} = Y\frac{3Flc^{1/2}}{2wt^2} \quad (1)$$

where $F$ is the load that caused the beam to

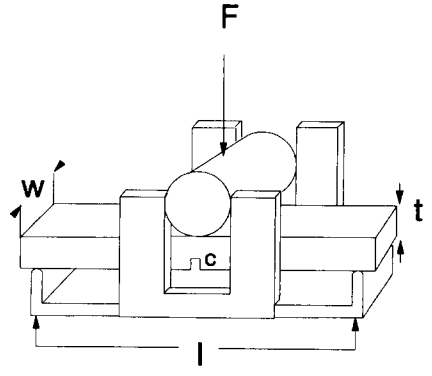


Fig. 1. Schematic diagram of the three-point single edged notched beam (SENB) test.

fracture, $c$ denotes the crack length, $l$ is the distance between rollers (17 mm), $w$ represents the width, $t$ is the thickness and $Y$ denotes a function of the geometry of the specimen expressed as a polynomial of the parameter $c/t$ (Brown and Srawley, 1966) given by Eqn 2:

$$Y = 1.93 - 3.07\left(\frac{c}{t}\right) + 14.53\left(\frac{c}{t}\right)^2 - 25.11\left(\frac{c}{t}\right)^3 + 25.8\left(\frac{c}{t}\right)^4 \quad (2)$$

The effect of specimen dimensions and crack lengths on the determination of critical stress intensity factor using the three point loaded single edge notched bend (SENB) test has been well studied both for ceramics (Bansal and Duckworth, 1980) and polymers (Hashemi and Williams, 1984) and has been the subject of an ASTM report (Brown and Srawley, 1966). The test has been criticised because of the difficulties in inserting sharp cracks into the specimens (Freiman, 1983) and assumes that sharp cracks will 'pop in' * at the notch during loading. Ceramics, described as being brittle, tend to be tougher and have higher breaking strengths than porous pharmaceutical specimens, i.e., pharmaceutical materials have critical stress intensity factors of the order 0.05–0.5 MPa $m^{1/2}$ whereas in general ceramics tend to be 0.5–18 MPa $m^{1/2}$ (Atkins and Mai, 1985). For low strength materials, e.g., pharmaceuticals, few problems are anticipated since, in contrast to polymers, whatever size of specimen or mode of loading, fracture will be brittle with little or no plasticity at the crack tip (Hashemi and Williams, 1984). The effect of plastic deformation is often represented by the size of the plastic zone near to the crack tip, and in brittle materials this tends to be small. The toughness of a material is best characterised by its value under plane-strain conditions, $K_{IC}$, and to achieve this the plastic zone must be appreciably less than the specimen thickness. This has been embodied in the ASTM empirical criterian for bend test (Brown and Srawley, 1966) such that:

$$c(\text{crack length}) > 2.5\left(\frac{K_{IC}}{\sigma_y}\right)^2 \quad (3)$$

$$t(\text{specimen thickness}) > 2.5\left(\frac{K_{IC}}{\sigma_y}\right)^2 \quad (4)$$

$$w(\text{specimen width}) > 5.0\left(\frac{K_{IC}}{\sigma_y}\right)^2 \quad (5)$$

where $\sigma_y$ is the yield stress.

With these equations it is possible to calculate the approximate physical dimensions of a specimen that will satisfy the criteria. For example, in the case of microcrystalline cellulose ($K_{ICo} \approx 0.7$ MPa $m^{1/2}$ (York et al., 1990) and $\sigma_y \approx 50$ MPa (Roberts and Rowe, 1987), the specimen size should be greater than 0.6 mm for crack length and thickness respectively, and 1.2 mm for the width. This concept has been embodied in the test implemented below.

## Materials and Methods

Microcrystalline cellulose (Avicel PH101, FMC Corp.) was used to validate the technique since it is a well characterised material in terms of its critical stress intensity factor, $K_{IC}$. In fact the same batch of microcrystalline cellulose was utilised as that tested by York et al. (1990), allowing direct comparisons between the three-point and four-point loading configuration to be made.

Other materials that were studied were as follows: anhydrous $\beta$-lactose (Sheffield Chemical Co.), $\alpha$-lactose monohydrate (extrafine grade, Dairy Crest), sucrose (icing grade, British Sugar),

* Note: 'pop-in', refers to method of introducing a sharp crack into the specimen. This is done by first generating a starter crack, which is then allowed to propogate before being stopped when of sufficient length of removal of the load (see Mashadi and Newton, 1988; and Roberts and Rowe, 1989.

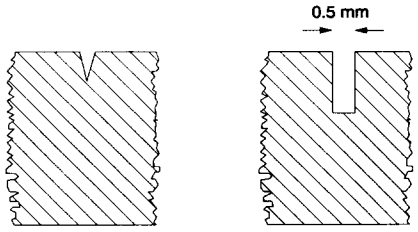


Fig. 2. Schematic diagram of the two types of notches.

adipic acid (BDH Chemicals Ltd), sodium chloride (micronised, ICI Chemicals and Polymers), Paracetamol DC (Graesser Salicylates), aspirin (hammer milled, Fluka chemicals), paracetamol (Thornton and Ross) and ibuprofen (Bradford University).

Beams of dimensions (20 mm × 7 mm × $t$), where $t$ was approximately between 3 and 4 mm, were prepared using a small punch and die using about 500 mg material compressed at varying compaction loads on a Tensometer (M30K, J.J. Lloyd) or a hydraulic press (Specac). The beam thickness, $t$, was greater than those used for modulus measurement (Roberts et al., 1991) to enable notches to be included. For microcrystalline cellulose a comparison was made between two notch types – the small 'V' notch (Fig. 2) and the preferred sawn slit notch (Fig. 2) to investigate the effect of notch depth and shape on the critical stress intensity factor. The V notch was produced by pressing a razor blade into the beam and sawn notches, essentially slits of width 0.5 mm (Fig. 2), were prepared by using a small saw blade (Agar Scientific).

Notch lengths (crack length, $c$) were measured using a travelling microscope to an accuracy of ±0.05 mm. The beams were placed on the three point bend rig as shown in Fig. 1 and stressed using a Tensometer at a constant velocity of 0.2 mm $min^{-1}$. The load, $F$, that caused the beam to fracture was recorded and used to calculate the critical stress intensity factor of the beam specimen using Eqn. 1.

The critical stress intensity factor at zero porosity, $K_{ICo}$, was evaluated by extrapolating the specimen $K_{IC}$ at porosity, $P$, using an exponential equation as suggested by Roberts and Rowe (1989) and York et al. (1990):

$$K_{IC} = K_{ICo} \exp^{-bP} \quad (6)$$

where $b$ is a constant.

## Results and Discussion

*Validation of the method using microcrystalline cellulose*

In a recent paper, York et al. (1990) examined the effects of notch shape on the critical stress intensity for beams of 10% porosity of microcrystalline cellulose and showed that for V shaped notches there was more variability when compared against straight notches when the dimensions of the notch were varied. Furthermore, the original test introduced by Mashadi and Newton (1987b) used V notches in a four-point bend configuration. It is therefore important that the effect of notch shape is investigated more fully and that the proposed three point test is validated against the four point loading configuration.

A comparison between results from this test and those of York et al. (1990) for the four point notched beam (notch length = 1 mm and width = 0.1 mm) is shown in Fig. 3, as a plot of measured $K_{IC}$ vs the porosity of the beams. Regression

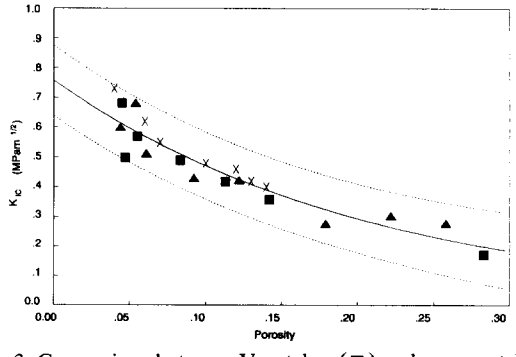


Fig. 3. Comparison between V notches (■) and sawn notches (▲) for the three-point SENB test and the four-point SENB results (×) of York et al. (1990), where the dotted line represents the 95% confidence limits and the full line the best fit for the data points using the exponential relationship (Eqn 6).

analysis was performed on each individual set of results and the constants, correlation coefficients and standard errors presented in Table 1. The values of the critical stress intensity factor at zero porosity, $K_{ICo}$, for the two crack geometries in the three point bend test show similar variability as evident from the standard errors (Table 1). Furthermore, the blunter sawn notch test has a lower $K_{ICo}$ than the sharper V notch.

Despite the fact that the four point test has a higher $K_{ICo}$ possibly due to the fact that the notch width is smaller (0.1 mm) as compared to the three point bend test (0.5 mm), when all the data are combined the results from each test lie extremely close to the mean line (Fig. 3) and hence the small differences seen may be due to the fact that for the four point SENB test measurements were only taken over a limited part of the porosity range.

Examination of the crack propagation through two beams of the same porosity (Fig. 4a and b) shows similarities in the shape of the crack path which is not straight. It is interesting to note that in both crack geometries the crack has been deflected due to the influence of the central roller but later appears to revert to its original path. It may well be that the cracks that run horizontally along the length of the beam (Fig. 4b), i.e., at right angles to the direction of the compaction load, have some influence on the vertical cracking. It is interesting to note that the introduction of weak interfaces into a ceramic can increase the toughness of brittle materials by deflecting a growing crack (Clegg et al., 1990) and this may be an important factor influencing the toughness of microcrystalline cellulose specimens.

$K_{ICo}$ determined using the three-point SENB test for microcrystalline cellulose can be compared to that from the four-point SENB test used by Mashadi and Newton (1987b) since a V notch was utilised in both cases. They reported a value of 1.21 MPa $m^{1/2}$ for a notch of depth 0.5 mm

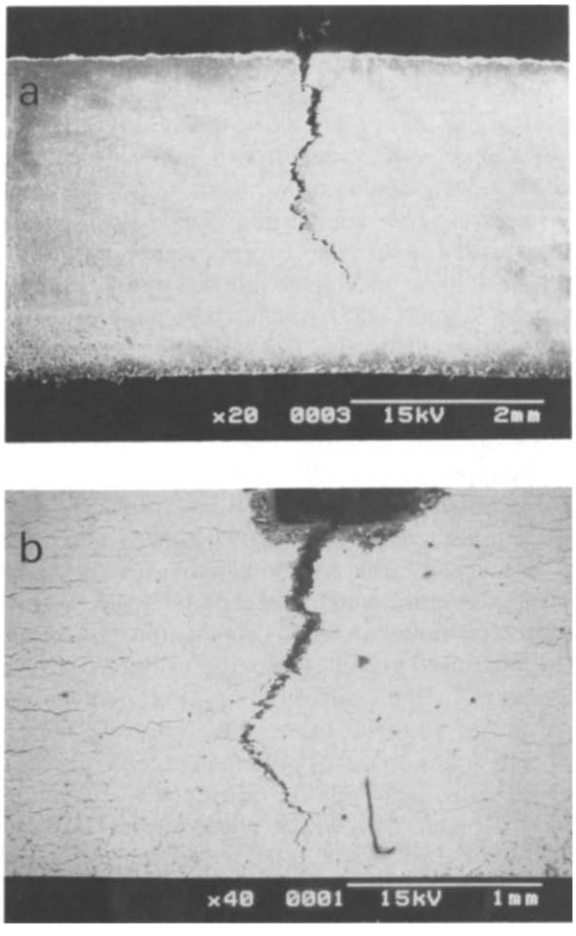


Fig. 4. (a) Scanning electron photomicrograph (SEM) of a V notch in a beam of microcrystalline cellulose; (b) SEM of a sawn notch in a beam of microcrystalline cellulose.

TABLE 1

*Regression analysis results for microcrystalline cellulose*

| Regression analysis results | Three-point 'V' notch | Three-point Sawn notch | Four-point straight notch | Combined data |
|---|---|---|---|---|
| $K_{ICo}$ (MPa $m^{1/2}$) | 0.7519 | 0.6927 | 0.8675 | 0.7568 |
| $b$ | 5.2486 | 3.9897 | 5.5907 | 4.7415 |
| Correlation coefficient | 0.9813 | 0.9307 | 0.9836 | 0.9474 |
| Standard error | 0.0555 | 0.0630 | 0.0253 | 0.0529 |

being somewhat higher than for the test here with a value of 0.75 MPa $m^{1/2}$ for a 0.9 mm notch depth. Additionally, Mashadi and Newton (1987b) used a glass cutter to score a notch into the beam which may produce a sharper crack than those in this study and it is known that for blunt crack systems the true equilibrium crack length before fast brittle fracture may be underestimated resulting in a lower calculated critical stress intensity factors (Munz, 1983). Similarly techniques involving test geometries utilising sharper cracks tend to give much higher fracture toughness values, e.g. double torsion, $K_{ICo} = 1.81$ MPa $m^{1/2}$ (Mashadi and Newton, 1988) and radially edge cracked disc ($K_{ICo} = 2.24$ MPa $m^{1/2}$ (Roberts and Rowe, 1989) (see table 2). It has also been suggested that microcrystalline cellulose is a material with a rising crack resistance curve (York et al., 1990). As crack lengths becomes longer there is more resistance to crack propagation and hence the measured critical stress intensity factor rises (Munz, 1983). Furthermore, $K_{ICo}$ cannot be regarded as a unique material constant but is simply one point on a crack growth curve. York et al. (1990) also suggested that if a definitive value of $K_{ICo}$ is required individual crack resistance curves should be investigated to allow all materials to be compared under the same conditions. It is interesting to note that York et al. (1990) reported an increase in $K_{ICo}$ as particle size decreased for various grades of Avicel and noted that similar effects occur in ceramic materials. This effect is probably due to an increase in the number of contact points within the porous specimens as particle size decreases.

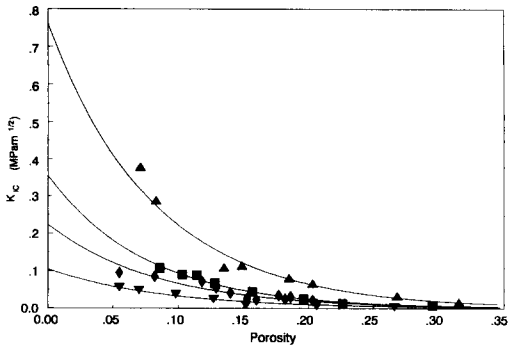


Fig. 5. Critical stress intensity factor vs porosity for various materials: (▲) anhydrous β-lactose, (■) α-lactose monohydrate, (◆) sucrose, (▼) ibuprofen.

*The critical stress intensity factor of excipients and drugs*

The results of $K_{IC}$ vs porosity for a number of selected excipients and drugs are shown in Fig. 5 with the line for the fit of the experimental points corresponding to the exponential relationship (Eqn 6). Data for the regression analysis of the results of all the materials are shown in Table 3, with both the correlation coefficients and standard errors of the results. The correlation coefficients and standard errors for the majority of materials tested were of a reasonable order. However, the correlation coefficient for paracetamol was low suggesting the influence of specimen

TABLE 2

*The effect of crack sharpness effects on the measurement of the critical stress intensity factor, $K_{ICo}$ of microcrystalline cellulose from various workers*

| Test | Notch type | $K_{ICo}$ (MPa $m^{1/2}$) | References |
|---|---|---|---|
| Radially edge cracked disc (compression) | pop-in | 2.98 | Roberts and Rowe (1989) |
| Radially edge cracked disc (tension) | pop-in | 2.24 | Roberts and Rowe (1989) |
| Double torsion | pop-in | 1.81 | Mashadi and Newton (1988) |
| Four-point SENB | V | 1.21 | Mashadi and Newton (1987b) |
| Four-point SENB | straight [a] | 0.87 | York et al. (1990) |
| Three-point SENB | razor | 0.76 | This work |
| Three-point SENB | sawn | 0.69 | This work |

[a] Straight-through notches cut using a lathe, e.g., same shape as sawn notches.

TABLE 3

*Critical stress intensity factors and regression analysis results (exponential equation) for various excipients, (where CC is the correlation coefficient and SE is the standard error)*

| Material | $K_{ICo}$ (MPa $m^{1/2}$) | $b$ | CC | SE |
|---|---|---|---|---|
| Anhydrous β-lactose | 0.7597 | 12.2736 | 0.9910 | 0.0295 |
| Avicel PH101 | 0.7569 | 4.7315 | 0.9474 | 0.0529 |
| Sodium chloride | 0.4769 | 7.2438 | 0.9487 | 0.0662 |
| α-Lactose monohydrate | 0.3540 | 13.2881 | 0.9895 | 0.0055 |
| Paracetamol DC | 0.2463 | 7.7112 | 0.9597 | 0.0197 |
| Sucrose | 0.2239 | 12.1346 | 0.9133 | 0.0107 |
| Aspirin | 0.1561 | 9.5791 | 0.9431 | 0.0087 |
| Adipic acid | 0.1398 | 11.1476 | 0.9602 | 0.0086 |
| Paracetamol | 0.1153 | 6.8934 | 0.8865 | 0.0140 |
| Ibuprofen | 0.1044 | 11.4747 | 0.9635 | 0.0049 |

effects, e.g., the presence of microcracks in the beam specimens.

It would be useful to compare values of critical stress intensity factor of these materials to values reported in the literature. However the only available data are those from microindentation testing (Duncan-Hewitt and Weatherly, 1989b) who reported values for sodium chloride, sucrose, paracetamol and adipic acid of 0.50, 0.08, 0.05 and 0.02 MPa $m^{1/2}$, respectively. These with the exception of sodium chloride are in general much lower than the values measured in this work (Table 3). Furthermore, the value for adipic acid is, in terms of rank order, lower by indentation than by SENB testing. These findings are not unexpected since the indentation technique has been criticised by a number of workers, notably Ponton and Rawlings (1989a, b), who commented that, in general, the microindentation test tends to underestimate values of the true material fracture toughness, because of slow post indentation crack growth. However the same authors considered that the SENB test gives a value which tends to be overestimated due to fast cracking. A detailed comparison between the two techniques is beyond the scope of this paper but will be considered in a future paper.

It is interesting that anhydrous β-lactose has a critical stress intensity factor about twice the value of α-lactose monohydrate. Similarly differences of the same order has been reported for measurements of indentation hardness for these two materials with values of 251 and 515 MPa, respectively (Leuenberger, 1982). The critical stress intensity factor of the carbohydrates can be compared with that of sorbitol instant (Mashadi and Newton, 1987a) with a reported value of $K_{ICo}$ = 0.47 MPa $m^{1/2}$. The carbohydrates can thus be arranged in the following order to decreasing $K_{ICo}$, anhydrous β-lactose > sorbitol instant > α-lactose monohydrate > sucrose.

The critical stress intensity factor as well as describing the ability of a material to resist fracture can be thought of as representing the stress to cause failure and therefore an indication of whether a material is weak or strong. Low values of critical stress intensity factor represent materials which are inherently weak, e.g., paracetamol and ibuprofen and high values represent materials which are strong, e.g., anhydrous β-lactose and microcrystalline cellulose. This definition has similarities to Hiestand's measure of brittleness, the brittle fracture propensity (BFP), a measure of the ability of a material to relieve stress (Hiestand et al., 1977), since it was suggested that a high BFP might indicate a materials suceptibility to lamination and capping or low strength. Despite this, there are clear differences in the response to stress of ibuprofen and paracetamol as evident from literature data. In terms of ductility ibuprofen is much more ductile than paracetamol with reported yield pressures of 25 MPa (Bateman et al., 1987) and 99 MPa (Von Podczeck and Wenzel, 1989), respectively.

Finally it is interesting to note that Paracetamol DC (a spray dried material containing 4% hydrolysed gelatin) has a critical stress intensity factor twice as large as that of paracetamol drug. This would appear reasonable from the expected function of a binder which is to increase both the strength of bonds between particles and the overall ductility of the material reflected in an increase in the toughness.

*Brittleness index*

Although it is clear that ductility can be reflected by a measure of the yield stress or indentation hardness, the brittleness of a material re-

quires a further parameter to describe this type of mechanical reaction to stress. Lawn and Marshall (1979) considered that the brittleness of a material is a measure of the relative susceptibility of a material to two competing mechanical responses, deformation and fracture. The abrupt brittle/ductile transition of many materials as size is reduced (Roberts and Rowe, 1989a) is a classic manifestation of this competition. Lawn and Marshall (1979) suggested the used of the indentation test which gives both parameters describing the resistance to deformation and the resistance of a material to cracking, i.e. hardness, $H$, and the critical stress intensity factor, $K_c$, respectively. They proposed a simple index of brittleness and the ratio of hardness to toughness, $H/K_c$, with units $\mu m^{-1/2}$.

By reference to literature data of indentation hardness and critical stress intensity factors, $K_{ICo}$ from this study, the brittleness index, $H/K_{ICo}$ can be computed. These data are presented in Table 4 with additional values of $H/K_c$ from Duncan-Hewitt and Weatherly (1989b) for comparison. Duncan-Hewitt and Weatherly (1989b) used this brittleness index to indicate that sucrose, adipic acid and paracetamol (with high values) were materials which fragment extensively during compaction (Table 4) whereas sodium chloride with a value an order of magnitude smaller (Table 4) was indicative of a material which essentially behaves in a ductile manner during compaction. Although the absolute magnitude of a parameter $H/K_c$ from Duncan-Hewitt and Weatherly (1989b) does not agree with $H/K_{ICo}$ the rank order for the materials is the same. The difference in magnitude is probably due to the under-estimation of $K_c$ in the indentation test as mentioned above.

The most striking feature of this data is that in terms of the brittleness index the drugs ibuprofen and paracetamol are at opposite ends of the scale whereas in terms of their critical stress intensity factors they are very similar (Table 4). This suggests that although both materials are weak ibuprofen is more ductile than paracetamol. These assignments are in agreement with their compaction behaviour; ibuprofen is ductile but laminates extensively even at low compaction speeds (Bateman et al., 1987) but paracetamol is a highly brittle drug that laminates and caps (Shotten and Obiorah, 1975).

Similarly in the case of the brittleness index of the two lactoses. Although both are considered strong (Table 4) $\alpha$-lactose monohydrate is more brittle than anhydrous $\beta$-lactose.

It is useful to compare the values of $H/K_{ICo}$

TABLE 4

*The brittleness of materials as indicated by $H/K_{ICo}$ (where H is the indentation hardness (literature data) and $K_{ICo}$, is the critical stress intensity factor of the material) as compared to $H/K_c$ of Duncan-Hewitt and Weatherly (1989b)*

| Material | $H$ (MPa) | $K_{ICo}$ (MPa $m^{1/2}$) | $K_{ICo}$ ($\mu m^{-1/2}$) | $H/K_c$ ($\mu m^{-1/2}$) | |
|---|---|---|---|---|---|
| Avicel PH101 | 168 [a] | 0.7569 | 0.22 | – | |
| Anhydrous $\beta$-lactose | 251 [b] | 0.7597 | 0.33 | – | |
| Ibuprofen | 35 [b] | 0.1044 | 0.34 | – | |
| Sodium chloride | 213 [c] | 0.4769 | 0.45 | 0.4 | |
| Aspirin | 87 [d] | 0.1561 | 0.56 | – | |
| Adipic acid | 123 [c] | 0.1398 | 0.88 | 6.3 | *Increasing brittleness* |
| Paracetamol DC | 265 [a] | 0.2463 | 1.08 | – | |
| $\alpha$-Lactose monohydrate | 515 [b] | 0.3540 | 1.45 | – | |
| Sucrose | 645 [c] | 0.2239 | 2.88 | 8.3 | |
| Paracetamol | 421 [c] | 0.1153 | 3.65 | 8.3 | |

[a] Jetzer et al. (1983).
[b] Leuenberger (1982).
[c] Duncan-Hewitt and Weatherly (1989b).
[d] Ridgway et al. (1969).

from this work with those of $H/K_c$ from Lawn and Marshall (1979) since it is important that pharmaceutical materials are compared with other materials based on this brittleness scale. The least brittle material, microcrystalline cellulose, is comparable with a value of 0.1 to medium strength steel (Lawn and Marshall, 1979) and 0.14 for polymethylmethacrylate, e.g., using $H =$ 210 MPa and $K_{IC} = 1.46$ MPa $m^{1/2}$ (Ritter et al., 1988). At the other end of the brittleness scale, paracetamol which is extremely brittle has a value comparable to that for $Al_2O_3$ ($H/K_c = 3$) but not as high as that for glass ($H/K_c = 8.8$).

Thus, in general, pharmaceutical materials cover a wide range of behaviour in terms of brittleness, from semi-brittle to brittle materials showing similarities to most classes of engineering materials, e.g., metals, polymers and ceramics.

## Conclusion

The technique of three point single edge notched beam (SENB) testing on pharmaceutical materials has been successfully validated using microcrystalline cellulose as a standard material. The $K_{ICo}$ values compare well with those determined by four point single edge notched beam testing and the technique has the added advantage that only a small amount of material is used.

The values of $K_{ICo}$ of a wide range of pharmaceutical materials determined by SENB testing are considered to be accurate and representative. Pharmaceutical materials including both drugs and excipients span a wide range of critical stress intensity factors ranging from 'weak' materials like paracetamol and ibuprofen to 'strong' materials like anhydrous $\beta$-lactose and Avicel. In terms of brittleness, the index $H/K_{ICo}$ is excellent in its ability to distinguish and describe the deformation behaviour of both excipients and drugs.

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