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Axial tensile fracture of microcrystalline cellulose compacts

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Abstract

An adapted tensile stress methodology for the fracture of microcrystalline cellulose (MCC) tablets has been investigated and implemented. The application of the generally applied linear elastic fracture mechanic (LEFM) parameters used to describe the fracture behaviour of these porous systems has been discussed. The application of an effective crack length concept, comprising of the notch depth and a process zone length designated Δc, has enabled the localised non-linear response of the MCC tablets to be characterised in a quantified manner. The requirement of the composite value Δc is postulated to be a direct result of the internal properties of the tablet formed during the compaction process due to its strong dependence on porosity. The high compact relative density creates a greater possibility for both local small-scale plastic yielding at the crack tip, commonly found in polymer materials and microcracking ahead of the crack tip, typically observed in the fracture of ceramics. The extrapolated value of K_{IC0} of 0.72 MPa $m^{0.5}$ found in this work lies within the range found in literature for this material indicating that the adopted procedure is acceptable for the determination of the resistance to fracture of MCC compacts.

Keywords: Microcrystalline cellulose; Fracture mechanics; Effective crack length; Tensile strength

1. Introduction

The manufacture of tablets of powder compacts of MCC within the pharmaceutical industry is of key importance as it provides the commonly used method of active ingredient dosage: oral ingestion. The characterisation of powder compacts through mechanical testing provides crucial information for the development of the compaction and processing stages of tablet production. The manufacture of multilayer tablets within the pharmaceutical tabletting industry has been available for at least the last 50 years (Cleaver, 1969; Little and Mitchell, 1949) with increasingly more complex designs and formulations being created. The sequential compaction of powder layers has enabled tailored release profiles of the active ingredient to be obtained (Abdul and Poddar, 2004). The multilayer formulation has however also created new challenges for the mechanical strength testing methodologies. Bilayer tablets have shown to catastrophically delaminate at the non-planer interface between the adjacent compacted layers (Inman et al., 2007). The fracture process that occurs is thought to be caused by internal tensile stresses normal to the plane of fracture as found during the fracture of single compacted tablets during capping (Nystrom et al., 1978). The experimental quantitative strength determining fracture process of the interface of bilayered tablets has therefore created a demand for a mechanical fracture methodology where a tensile stress is developed orthogonal to the interface between adjacent compacted layers, along the axial plane. Commonly applied methodologies for the determination of the relative strength or resistance to fracture of materials include indentation (Miyazaki et al., 2007), three- and four-point beam bending (Mashadi and Newton, 1987; York et al., 1990; Roberts et al., 1993; Hancock et al., 2000), deeply double edge notched tension (Gong et al., 2005), double torsion (Mashadi and Newton, 1988), radially edge cracked disks (Roberts and Rowe, 1989; Kendall and Gregory, 1987), diametrical compression (Mohammed et al., 2005; Rudnick et al., 1963) and friability testing (Shepler and Whitney, 1978; Riippi et al., 1998; Schultz and Kleinebudde, 1994). These fracture methods, however, either require the production of specimens in unique geometries which are not found within industrial processing, or do not result in an applied tensile stress acting in the axial plane of a cylindrical compact. A

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fracture methodology and associated analysis that seems to have been overlooked in recent times is the axial tensile test developed by Nystrom et al. (1977). During testing a tensile stress develops which is normal to the axis of the tablet and hence has shown a good correlation with the observed capping tendency of different pharmaceutical materials (Nystrom et al., 1978). Karehill et al. (1990) have shown that the fracture of bilayered pharmaceutical tablets can be achieved using this methodology when investigating the effects of surface roughness on adhesion. However no substantial investigation into the mechanics of fracture was undertaken.

2. Fracture mechanics

The theoretical study and application of the mechanics of crack growth within pharmaceutical materials is important as it helps to more fully characterise the failure process of these tablets and may elucidate possible solutions to prevent tablet cohesive failure within future manufacturing. The porosity of powder compacts makes the prediction and measurement of tablet strength relatively complex: inherent space within the compact creates the possibility of strength weakening internal flaws or cracks. Due to crack propagation the bonded particles separate sequentially (Kendall, 1988), meaning the application of a Griffith (Griffith, 1920) style energy balance is appropriate. Griffith theory, the basis for linear elastic fracture mechanics (LEFM), assumes that the deformation at a crack tip within a material is completely elastic and thus no plastic deformation of the material occurs. The experimental determination of LEFM parameters used to describe the material properties of MCC during fracture has been published by various authors (Mashadi and Newton, 1987, 1988; Roberts and Rowe, 1989; York et al., 1990; Roberts et al., 1993; Hancock et al., 2000). Within these publications there has been no attempt to introduce the application of non-linear fracture mechanic principles to account for the small zone (ca. 1 mm, for example) of plastic deformation ahead of the crack tip, which is surprising given the inherent ductile nature of these materials. MCC compacts are consolidated particulate solids made of cellulose polymers and therefore it would seem logical that the fracture behaviour of MCC tablets is a combination of the characteristic behaviour of both ceramic fracture (brittle and unstable) and polymer fracture (displaying relatively small scale local plastic yielding). Thus the LEFM assumption of a fully elastic response at a sharp crack tip appears to be an erroneous simplification of a more complex fracture propagation process. Small scale plastic yielding at a crack tip does not automatically mean that LEFM cannot be applied, merely a 'modified', as opposed to an apparent, crack length must be included in the fracture parameter calculations (Irwin, 1957, 1961) to fully understand the material response of MCC compacts to a propagating crack. The commonly quoted LEFM parameter K_C or critical stress intensity factor provides an indication of the brittleness of a material by evaluating the stress field near a crack tip. As discontinuities such as pores or flaws, of a variety of sizes and shapes, will exist in the material concentrating the local stresses, K_{IC} is usually obtained from the fracture stress of a sample containing a significantly dominant pre-notch or crack of known length (Eq. (1)).

$$K_C = Y\sigma c^{0.5} \tag{1}$$

where Y is a polynomial function based on the geometry of the sample and loading, σ the fracture stress and c is the crack length. When relatively small scale crack yielding is present, c is arbitrarily replaced by an effective crack length $\hat{c}$ (Eq. (2)) where the crack length has been extended by the radius of a supposed process or inelastic zone Δc.

$$\hat{c} = c + \Delta c \tag{2}$$

The exact interpretation of the process zone is fairly ambiguous. However, it is well understood that at least for ductile materials the process zone is a key parameter that prescribes the toughness (Adams et al., 1989) and therefore seems to be a logical inclusion for the fracture parameter calculations for MCC. The use of LEFM to characterise the fracture behaviour without this process zone extension merely provides a quantitative 'estimate' of the fracture mechanism, suitable for general comparison and the ranking of materials. The main purpose of this current work is to develop the analysis of the MCC compacts material response to fracture utilising an axial tensile stress methodology. By computationally including any process zone, which may be present at the crack tip it is hoped a more rigorous analytical approach to the fracture of these porous bodies will improve the understanding of the mechanical response to fracture of MCC compacts.

3. Experimental procedure

3.1. Materials

Microcrystalline cellulose (MCC) was used as supplied in the form of Avicel PH102, FMC Corp., Philadelphia, USA. The material has a mean particle size 95.3 μm averaged over three runs using a HORIBA LA-950 Wet ver. 3.2 particle seizer (Particle Technology Ltd., Derbyshire, UK). The samples were dispersed in deionised water and no wetting agent or surfactant was used. No ultrasonics was applied to provide the closest possible representation of the ambient dry powder properties. Preliminary runs of the sample initially after wetting and after a period of 5 min showed that no swelling of the particles occurred. MCC has a true (pycnometric) density of 1577 kg/m^3 (obtained from the average of 10 runs using an AccuPycnometer 1330, Micromeritics, Bedfordshire, UK). MCC is a largely ductile material and hence deforms in an extensive plastic manner under a compressive stress (Roberts and Rowe, 1987).

3.2. Compaction

An unlubricated flat-faced punch and die set was utilised, which was manufactured from optically polished hardened stainless steel supplied by Specac Ltd., Kent, UK. The diameter of the die was 20 mm producing tablets of 20 mm diameter compressed to a maximum pressure of between 12.7–156 MPa. The weight of the samples was varied to provide a constant aspect ratio of the tablets of 0.5 (final compact height of 10 mm). Variability within