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# Letter to the Editor

# Brittle–Ductile Transitions in Organic Solids During Comminution: a Practical Demonstration

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Pharmaceutical materials vary from those that are brittle and consolidate by fracture or fragmentation (e.g. the inorganic carbonates) to those that are ductile and consolidate by plastic deformation or flow (e.g. microcrystalline cellulose). However many materials consolidate by both fragmentation and plastic flow, the dominant mechanism depending on their particle size. The particle size at which the behaviour changes is termed the brittle–ductile transition (Kendall 1978). This behaviour manifests itself in a hammer mill as the size at which it is impossible to comminute particles by compression induced by the hammers.

Kendall (1978) has shown that it is possible to predict the critical size at the brittle–ductile transition, $d_{crit}$, by use of the expression:

$$d_{crit} = (AK_{IC}/\sigma_y)^2 \qquad (1)$$

where $K_{IC}$ and $\sigma_y$ are the critical stress-intensity factor and the yield stress, respectively, of the material and A is a constant equivalent to $(32/3)^{0.5}$ or 3·27 for rectangular particles.

Brittle–ductile transitions have now been predicted for a wide range of pharmaceutical materials and are in good agreement with experimental measurements (Roberts & Rowe 1987; Rowe & Roberts 1995). All that is required is independent measurement of $K_{IC}$ and $\sigma_y$. This communication presents a practical demonstration of the concept in the hammer milling of an organic solid, a dipeptide dihydrate consisting of needles with a high aspect ratio.

The yield stress of the material was measured at a punch velocity of 0·033 mm $s^{-1}$ by means of a compression simulator as described elsewhere (Roberts & Rowe 1985) and found to be 103·1 ± 2·2 MPa. The actual critical stress-intensity factor was measured by means of three-point single-edge notched beam testing as described elsewhere (Roberts et al 1993) and found to be 0·3613 ± 0·0324 $MPam^{\frac{1}{2}}$.

Substituting these values into equation 1 generates a brittle–ductile transition of 131 $\mu$m with a range of 104–160 $\mu$m. Of course, these are predictions for $d_{crit}$ at low strain rates and it is known that increasing strain rates tend to cause a slight decrease in the transition (Roberts et al 1989). Hence for hammer milling at high strain rates it would be expected that $d_{crit}$ would be (approx.) 100 $\mu$m.

Needle-length distributions measured by image analysis (Quantimet 520, Cambridge Instruments, Cambridge, UK) for three batches of the material, unmilled and milled are shown in Figure 1. For the two batches where there is an insignificant number of needles greater than 100 $\mu$m there is no difference between the needle-length distributions before and after milling, i.e. the unmilled crystals are below the brittle–ductile transition, therefore no comminution has taken place. However, for the batch of which 10% (approx.) of the needles are > 100 $\mu$m these are fractured in the mill producing smaller needles and the needle-length distribution tends towards that of the other two batches. This suggests that the brittle–ductile

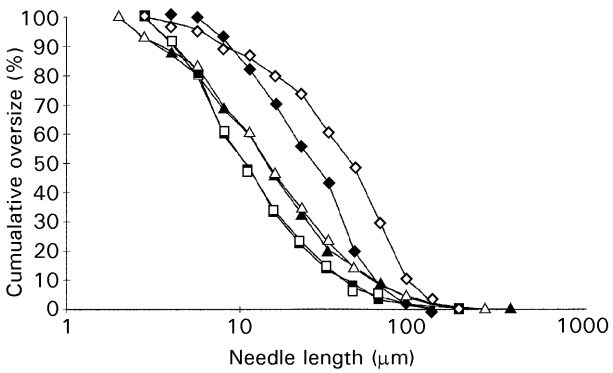


Figure 1. Needle-length distributions of three batches of drug before (□, △, ◇) and after (■, ▲, ◆) milling.

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transition is at 100 $\mu$m (approx.), similar to that predicted.

The results show the benefit of measuring the mechanical properties of new drugs when predicting their behaviour in hammer mills. In addition the techniques are economical in their use of drug, requiring a total of 3 g to generate the data used in this study (2 g for single-edge notched beam testing, 1 g for compression simulator).

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