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Effects of punches with embossed features on compaction behaviour

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ABSTRACT

This work investigated the modifications of compaction behaviour that resulted from using punches with one or two embossed (i.e. raised) ridges across their contact faces, to produce debossed furrows on otherwise flat-faced cylindrical specimens. Single-sided uniaxial compaction of spheronised microcrystalline cellulose was used to prepare specimens of different thickness and density. Based on methods developed previously, small-angle X-ray scattering (SAXS) was used to map local density variations, compressive stress, strain and strain direction over diametral sections from the specimens. The results revealed zones of relatively high compaction below the debossed furrows, with lower compaction along the flanks. High compressive stresses (up to eight times the average peak punch pressure) were indicated just below the furrows, which decreased exponentially with increasing distance below the furrows. Extrapolation towards the surface suggested that even higher stress occurred where the powder was in direct contact with the embossed ridges on the punches. It is suggested that the significant variations in relative density, stress, strain and principal strain direction observed around the debossed features could contribute towards the pressing faults that are sometimes encountered during commercial tabletting. The high stresses may also cause crystallographic changes in susceptible materials. Moreover, although the present work was performed using a pharmaceutical material, it seems likely that similar effects would also arise where powder compaction is used to produce metallic or ceramic items with complex shape.

1. Introduction

Consolidation of powders under pressure between punches in a die is widely used to manufacture products from various polymer, ceramic and metallic materials [1]. Powder compaction can be particularly effective in forming objects of complex shape (e.g. tools, gear wheels and other engineering parts), where melt-processing is not possible (e.g. ceramics with high melting points or materials that decompose at elevated temperatures), where the material is too hard to permit other processing routes (e.g. manufacturing cutting tools) or where controlled porosity is advantageous (e.g. filters, catalyst supports and 'self-lubricating' bearings). Indeed, in many cases, powder compaction can provide multiple advantages over other manufacturing methods.

The pharmaceutical industry uses powder compaction extensively to produce tablets, which remain a very popular and widely used dosage form [2–4]. The manufacture of tablets of complex shape (incorporating debossed break lines, logos and other features), of precise size and composition (to ensure accurate dose levels), without excessive heating (to avoid decomposition), with minimal wastage (of expensive and potentially harmful drugs) and controlled porosity (to ensure a suitable balance between adequate strength and disintegration behaviour) is a clear demonstration of the multiple advantages that powder compaction can deliver, compared with other processing routes. Moreover, as tabletting usually involves combinations of drugs, excipients and processing conditions, it has inspired much work to explore the relationships between materials properties, compaction behaviour and the resulting tablet characteristics.

An important and widely studied aspect of powder compaction is that it generally results in local density variations within the articles produced. Although earlier reports can be found, the first systematic investigation was by Train [5], using coloured layers to reveal differences in movements within the powder bed during compaction. Subsequent investigations have used a wide range of methods, including: further applications of the coloured layer method [6,7], local hardness measurements [8], autoradiography of radioactive specimens [9], nuclear magnetic resonance imaging (MRI) after infiltration with a suitable non-swelling liquid [10,11], X-ray microtomography (XμT) [12–14], near infrared (NIR) spectral imaging [15] and small-angle X-ray scattering (SAXS) [16–20]. In addition, computational methods have been widely used to simulate compaction behaviours for different combinations of materials, tooling geometry and process conditions, as reported in numerous research papers [19–29] and reviews [30–32].

In the simplest case of flat-faced cylindrical specimens produced by single-sided compaction (i.e. the compaction force is applied through only one driven punch), the most extreme variations occur around the circumference, with the highest density adjacent to the driven punch and lowest density adjacent to the static punch [1–3,5–10,13–17, 19,21,22]. This is due to friction against the tooling, which resists the movement of granules along the die walls [7,14,22]. Smaller variations occur within the compact, due to internal powder flows compensating for the restricted movement along the die walls, giving a high density

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zone just above the static punch and a low density zone below the driven punch.

The density variations depend on various material properties and processing parameters. The effect of friction can be changed by lubrication [5,6,9,24,25,28], while differences have been observed between plastic and brittle materials [10]. Double-sided compaction (i.e. the force is applied through two driven punches) produces patterns with greater mirror symmetry between the top and bottom halves of the tablets [3]. Non-circular (e.g. elongated, capsule- or lozenge-shaped) die bodies give more complex patterns of density variations, without the rotational symmetry produced by cylindrical dies [4,11,12]. The patterns also change if curved punch faces are used [7,19,23,25,29]. Complex punch geometries incorporating large features produce dramatic effects [20], while even relatively small embossed motifs (e.g. to produce break lines, company logos or other identification marks) result in higher density ahead of the debossed features and lower density along the flanks [4,11,12].

The pattern of density variations is important for several reasons. Firstly, cohesive strength and hardness increase with the local density of the compacted material [1–4,8,13,19–21,25,28,29,33–35]; hence, lower density regions will be weaker and may be more prone to mechanical failure. With regard to pharmaceutical tablets, insufficient mechanical strength may lead to unacceptable damage during subsequent processing or storage. Secondly, there is evidence linking density variations to differences in elastic recovery, which may cause deformation and spontaneous crack formation [22, 23, 36–38]. In the case of tablets composed of poorly cohesive materials, this can manifest as lamination and capping. Thirdly, the density variations are associated with local differences in porosity, which can affect the swelling and disintegration behaviour of the tablet [2–4,39]. Fourthly, many APIs are known to be polymorphic, with the amorphous and different crystalline forms showing different rates of dissolution [40]. Hence, differences in compaction stress associated with the density variations could cause changes in crystal form, with adverse consequences for the tablet performance.

Notwithstanding the body of previously published work, many details of powder compaction remain poorly understood and there is a clear need for further investigations. In particular, the effects of tooling geometry and embossed features on punch surfaces merit further study. This is clearly important in pharmaceutics, where tablets are commonly made with different shapes or debossed features, in order to make swallowing easier, to reduce problems associated with capping or lamination, to provide an element of commercial discrimination and to aid identification. It is suggested that these effects are also of more general relevance, however, since compaction of metal and ceramic powders is widely used to manufacture objects of complex shape, where control of porosity, strength and dimensional stability are very important.

The work presented here used SAXS to investigate the effect of debossed furrows on density variations within what would otherwise be simple flat-faced cylindrical compacts. Two previous studies revealed large density variations around debossed lines and lettering on 'capsule-shaped' tablets [11,12], but without investigating the relationships to other tabletting parameters. The present work addresses some of these issues, by exploring the range and magnitude of density variations and corresponding stresses, for different compaction pressures and tablet thickness.

Studies of compaction behaviour using SAXS have been described in detail, in several previous publications [16–20]. Based on measurements collected at different points over a diametral cross-section prepared from the compacted specimen, SAXS is able to provide spatially-resolved, quantitative information concerning compression of the nanometre-scale intragranular morphology. This can be used to evaluate local variations in density, compressive stress, strain and principal strain direction. Amongst previous work, SAXS was used to investigate the effects of complex punch geometries, incorporating a relatively large embossed cone, cylinder or hemispherical dome [20]. By contrast, the present work investigates the effect of smaller features, typical of those commonly found on pharmaceutical tablets.

2. Experimental

This work was carried out at the University of Huddersfield, using methods essentially similar to those reported previously [16–20]. Slight differences in apparatus and experimental procedures, however, gave some improvements in the present work.

All experiments were performed using Celphere SCP100 (Asahi-Kasei Corp. Japan), a commercial grade of spheronised microcrystalline cellulose (s-MCC), which has been used in previous studies. The granule structure was examined by SEM, using a JEOL 6060 LV (JEOL Ltd. Tokyo, Japan), in high vacuum mode at 15 keV, after coating with Pd (ca. 20 nm). This revealed roughly spherical granules with a mean diameter of around 100 μm, as shown in Fig. 1. As reported previously [16,17], this material represented a good model system, since the roughly spherical particles avoided any possibility of preferential granule orientation during die filling.

2.1. Preparation of compacted specimens

Specimens for mapping experiments were prepared using a polished stainless steel die (circular cross-section, internal diameter d = 10.00 mm, Specac, Orpington, UK), with a flat-faced lower punch. A pre-weighed amount of s-MCC granules (0.29–0.65 g, depending on the intended specimen height) was poured into the die and loosely packed by gently tapping against the bench.

Different upper punches were used to give the desired contact surface geometries. A second flat punch gave standard flat-faced cylindrical

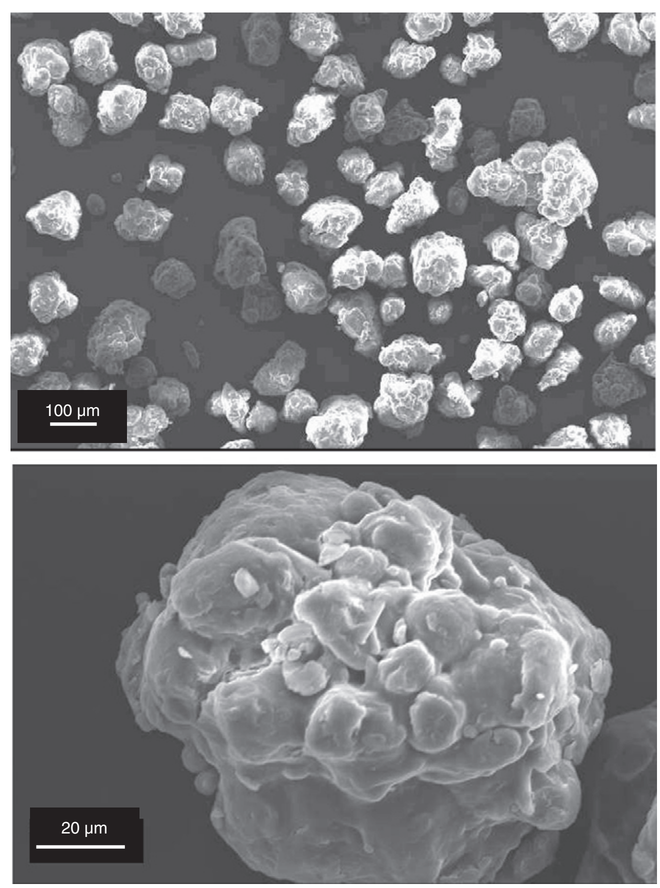

Fig. 1. SEM images of s-MCC.