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An apparatus for the study of strain recovery in compacts

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An experimental apparatus is described for use in determination of the strain recovery in compacts. The basic principle of the apparatus is the production of Fraunhofer diffraction fringes by two orthogonal slits formed between fixed edges and the edges of the compact, which permit small changes ($\sim 5 \times 10^{-5}$ m) in compact dimensions to be continuously monitored. One of the important advantages of the method is that it prodives a non-contact measurement and thus avoids damage to the compact surface which can arise through the use of micrometers, callipers or mechanical transducers. The whole apparatus, excluding the light source, may be fitted into an environmentally controlled cabinet, thus permitting temperature, humidity, etc. to be varied. Results are presented for the use of the apparatus in studying the strain recovery of methyl cellulose compacts.

1. Introduction

It is widely known in the pharmaceutical industry that both the mechanical properties and the dimensions of tablets can change significantly during storage. This occurrence is not only of fundamental scientific interest in that it reflects the rheological behaviour of compacts, but is also of commercial interest since during strain recovery it has been found that microcracks can develop which may ultimately result in the splitting of sugar coatings on commercial tablets [1].

One particular method of studying the rheological behaviour of compacts after the removal of compressive loading has been to observe the strain recovery [2]. Such observations show that the strain recovery is invariably a two stage process [3]. Immediately after release of the compressive forces the compact undergoes an instantaneous elastic recovery followed by a more gradual viscoelastic behaviour. This type of response is similar in many ways to the creep recovery of polymeric materials after tensile loading [4]. As yet no full physical explanation has been given for the viscoelastic behaviour of compacts. The inherent explanation must lie in the properties and molecular structure of the individual materials of which the compacts are composed and in the history of the processing of the compact. It has also been noticed that the effect of water sorption in compacts can cause swelling which can complicate studies of the viscoelastic relaxation process [1].

In this article the preliminary design of an optical instrument used for measuring dimensional changes in compacts is described. In particular this instrument has been used to investigate the effect of water sorption in compacts, causing swelling. This effect can be of prime importance in certain tropical countries where the humidity remains as high as 80 to 90% r.h. for six or more months of the year.

The few reported measurements of dimensional changes in in compacts [5, 1] have relied on the use of callipers, micrometers or linear transducers and have required physical contact with the

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surface of the compact. Repeated surface contact during measurements can lead to a marked deterioration of the compact surface causing inaccurate measurments. It is likely that in some instances the changes brought about by surface damage can nullify real changes brought about by strain recovery or swelling. In addition contact measurements are made with reference to an individual point on the compact's surface and to produce measurements over a wide area of the surface can be both laborious and time consuming.

The optical method described below is a non-contact method and therefore avoids surface damage of the compact. It also has the advantage that measurements can be made quickly and with ease whilst keeping the compact in a fixed environment.

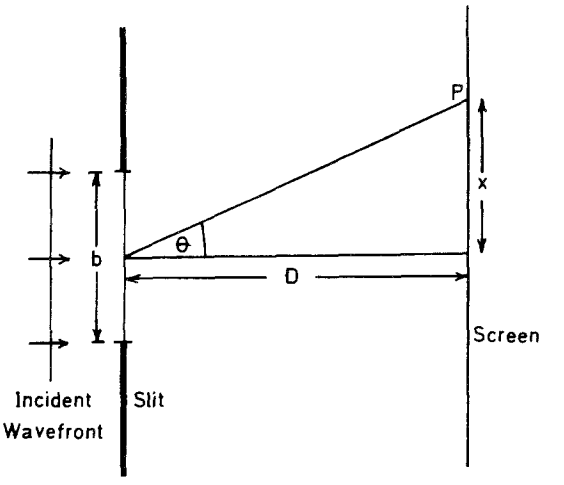

Figure 1 Experimental Fraunhofer diffraction arrangement.

2. Theory

The principle of the technique is to place the compact on some fixed surface in such a way that one edge and one face of the compact have their movement constrained. Close to the unrestrained edge and face and parallel with them are placed knife edges so as to form two orthogonal slits. The slits are in a plane lying along one diameter of the compact. The slits' width will vary as strain recovery occurs and the size may be determined by observing the diffraction pattern produced when the slits are illuminated with a monochromatic beam of light. If the distance between the knife edge and the fixed surface is known it is relatively simple to calculate the dimensional changes occurring.

The simplest experimental arrangement is to allow a parallel beam of monchromatic light to be incident normally on the slit and to observe the diffraction pattern on a screen set at a considerable distance behind the slit, as shown in Fig. 1. This type of diffraction is known as Fraunhofer diffraction. The development of the expression for the resulting light intensity I reaching a point P on the screen can be found in any undergraduate text on optics [6] and is given by:

$$I = A^2 = A_0^2 \frac{\sin \beta}{\beta} \tag{1}$$

where

$$\beta = \frac{\pi b \sin \theta}{\lambda}, \tag{2}$$

A is the amplitude of the diffracted wave reaching the screen at an angle θ to the normal. A_0 is the amplitude of the incident wavefront, b is the slit width and λ is the wavelength of the light used. As can be seen from the expression, the light intensity oscillates as θ varies, and minima occur when:

$$\beta = \frac{\pi b \sin \theta}{\lambda} = \pm n\pi, \tag{3}$$

where n is an integer. Rearranging Equation 3, b is obtained in terms of the variable θ:

$$b = \pm \frac{n\lambda}{\sin \theta}. \tag{4}$$

In general θ is small and it is, therefore, valid to put $\sin \theta = \theta$ and to write:

$$b = \pm \frac{n\lambda}{\theta}. \tag{5}$$

Also in practice it is easier to measure the distance x from the central maxima to the nth minima rather than the angle θ. If the distance of the screen from the slit is D then:

$$\theta = \frac{x}{D} \tag{6}$$

and

$$b = \pm \frac{n\lambda D}{x}. \tag{7}$$

Thus an expression for the slit width in terms of measurable quantities x and D is obtained.

It can also be shown that the standard error α_t in the determination of b is given by [7];

$$\alpha_b^2 = \left(\frac{n\lambda}{x}\right)^2\left[\alpha_D^2 + \left(\frac{D}{x}\right)^2\alpha_x^2\right] \quad (8)$$

where α_x and α_D are the standard errors in x and D respectively.

3. Apparatus

The main components of the measuring apparatus consist of a Moore and Wright micrometer screw gauge reading to ± 0.005 mm mounted vertically on an aluminium base plate, as shown in Fig. 2.

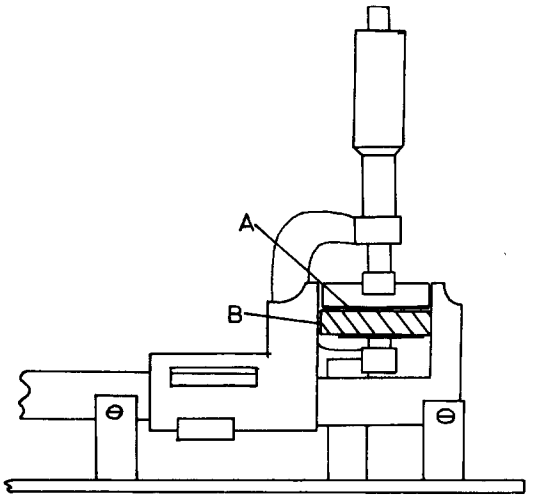

Figure 2 Micrometer and calliper mounting, showing the position of the compact with horizontal slit A and vertical slit B.

Also on the base plate and fixed horizontally, so that the centre line of the micrometer spindle and anvil lie in the plane of its jaws, is a vernier calliper. Attached to the micrometer anvil is a microscope coverslip on which the compact rests in such a way that the edge is also in contact with the fixed jaw of the callipers and the plane in which the jaws lie is along a diameter of the compact. Fixed to the micrometer spindle by a brass collar is a small stainless steel plate which forms the knife parallel to the coverslip on the anvil. This knife edge is used to produce a horizontal slit A of variable width with the top surface of the compact. The moveable jaw of the vernier, together with the edge of the compact, forms the vertical slit B.

The above mentioned part of the apparatus is positioned in a perspex cabinet, fitted with a close fitting door. This cabinet, which is used for environmental control, is mounted on a carriage fixed on an Ealing Optical optical bench. A Griffin and George 1 mW helium–neon laser (λ = 632.8 nm), also mounted on a carriage, fits onto the optical bench. The carriages used were of the type which permitted controlled vertical and horizontal movement with respect to the optical bed, measurable in 1 mm increments. By this means it was possible to position the compact such that the light beam could be moved in suitable increments across the two slits and the changes in dimensions of different parts of the compact monitored.

4. Precalibration and use of the apparatus

The optical bench was placed in such a manner that the diffracted light beam after leaving the slit traversed a distance of approximately 4 m before striking a screen. The screen consisted of several sheets of finely ruled graph paper with mm spacings, which facilitated the measuring of the spacing between the minima in the pattern. As can be seen from Equation 7, the slit width can be determined once x and D are known. It is a point of interest to note that it is possible to determine D ($\sim$ 400 cm) to an accuracy of ± 0.5 cm, whilst repeated measurements of x ($\sim$ 4 cm) showed a standard error of ± 0.1 cm. This means that in Equation 8 the first term is negligible compared to the second and the overall error on determining b is of the order of $\pm 6 \times 10^{-4}$ cm. To avoid the laborious technique of making repetitive calculations of b from x and D the following calibration technique was used. The micrometer was closed until the knife edge came into contact with the glass coverslip and the reading (a) noted. The micrometer was then opened to give a reading (c) and the distance $2x$ between the first two minima (n = 1) on either side of the central maxima was noted. The width of the slit causing the diffraction pattern was (c)–(a), the distance from the top of the coverslip to the knife edge. A series of readings were taken for different slit widths and a graph plotted of slit width against $1/2x$. This yielded a straight line as shown in Fig. 3.

To illustrate the use of the instrument, details are given below of the strain recovery of a methyl cellulose compact. A sample of methyl cellulose powder, Tylose MH 4000, obtained from Hoechst AG, Frankfurt, W. Germany was sieved to a size fraction 355 to 710 μm. The powder was then dried at 105° C for 48 h to a moisture content of

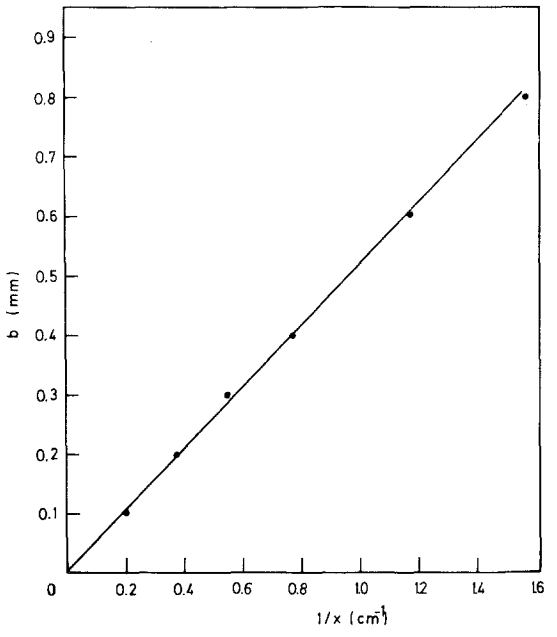

Figure 3 Calibration curve for the apparatus – the plot of b versus $1/2x$.

0.18% w/w and stored in a desiccator over silica gel. Compacts of this material were prepared using an Avery 30 ton type A804/1474 press with a loading of 100 MN m^{-2} and a 19.0 mm diameter die. The rate of loading, as measured using a Baty dial gauge, was 0.32 cm min^{-1}. Compacts were ejected from the die in the direction of compression at the same machine speed. At the end of the compression cycle the compact had a nominal diameter of 19.0 mm and a thickness of 5.73 mm. After release from the die the instantaneous changes in dimensions were determined by the use of vernier callipers. For the sample used, after ejection, the diameter was found to be 19.20 mm and the thickness to be 6.68 mm. This rapid increase in dimensions, equivalent to a 18.94% increase in volume, can be attributed to instantaneous elastic recovery. This compact was then mounted in the apparatus and the changes in dimensions at subsequent time intervals determined by observing changes in the diffraction patterns. Using the vertical and horizontal controls on the carriage on the optical bench, the surface of the compact was scanned at 4 mm intervals and three measurements were taken at 3 mm intervals down one edge. The relative humidity in the

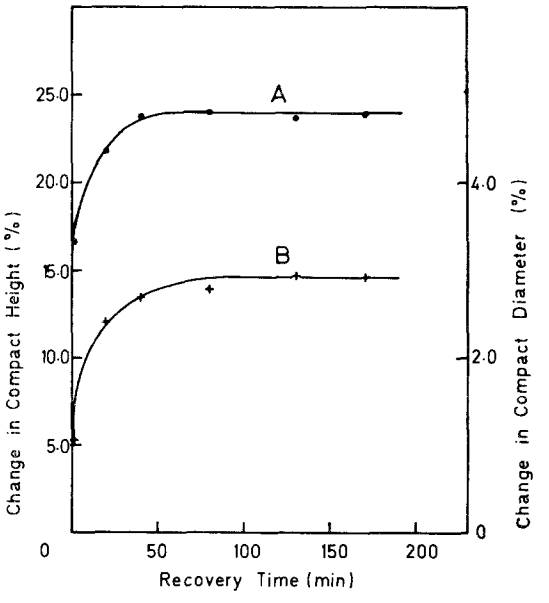

Figure 4 Strain recovery curves for the methyl cellulose compact; A, mean axial and B, mean radial changes in compact dimensions.

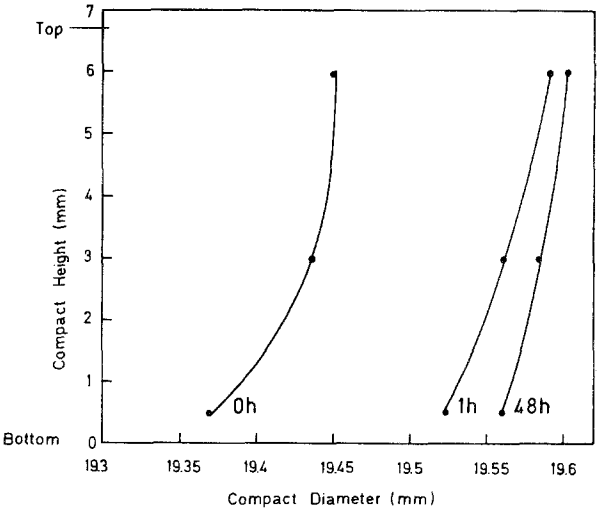

Figure 5 Change in edge shape of the compact during strain recovery at 0, 1 and 48 h after compact ejection from the die.

cabinet was maintained at 25% ± 5% using silica gel.

The mean axial (curve A) and radial changes (curve B) in the compact dimensions are shown in Fig. 4. The compact undergoes an immediate elastic recovery of 16.50% on the height and of 1.05% on the diameter. This is followed by a slower non-linear increase in dimensions until, after a period of approximately 100 min, only small changes in dimensions are recorded. Fig. 5 shows the change in shape of the edge of the compact with time as determined by measurements at three points along one edge. The measurements were made at 0, 1 and 48 h after ejection of the compact from the die. It is interesting to note that since the compact was compressed in a perfectly cylindrical die it is probable that the edge shape could be due to a non-uniform stress distribution within the compact or to sorption of surface moisture. The compact in its final state of equilibrium showed an increase in excess of 31% over the volume of the compact immediately prior to its release from the die. Further details of compact behaviour are to be published elsewhere [8].

5. Conclusion

The technique of Fraunhofer diffraction has been shown to be suitable for the use of determining the strain recovery in compacts. It has the following advantages:

(1) The technique is quick to use compared with the use of callipers or electromagnetic transducers. This is important when dimensional changes occur rapidly.

(2) Measurements can be made entirely within an enclosed area thus permitting close control of humidity, temperature or other environmental conditions.

(3) The method is of a non-contact type and hence avoids local damage or contamination of the surface of the compact.

(4) By suitable development it would be possible to devise a photoelectric detection system of the diffracted beam coupled with a digital readout. Also by use of additional micrometer-calliper sets and with suitable beam deflection, measurements could be made on more than one compact.

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