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The use of photoelastic techniques in the measurement of die-wall stress in tabletting

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A new technique is described for the observation of the stresses acting during a compaction operation. Pharmaceutical materials have been compressed in a Perspex die and viewed by polarised light. By using ancillary Perspex strain gauges both radial pressure and shear stress at the die wall may be determined from the interference pattern. Although Perspex is not a strong material, pressures up to about 12 tons/in^2 could be contained, and a hard tablet extracted from the die. The entire pressing operation is recorded on ciné film for later analysis.

VARIOUS authors have attempted to measure the variation of radial stress along the cylindrical surface of a compact, and to relate it to the gradual decay of pressure away from the face of the punch. Nelson (1955) has constructed a special punch and die assembly in which a movable section of the die wall is connected to a load cell. With this arrangement he was able to determine die wall pressure, not at a particular point, but only in a region. Windheuser, Misra, Eriksen & Higuchi (1963) made experiments in which a strain gauge was attached to the outside of the die in a zone where the wall thickness had been reduced sufficiently to allow deflection under radial pressure. We have considered the possibility of constructing a ring of piezo-electric material which would be recessed inside the die and would effectively form a part of the die wall. Radial pressure would then be converted into a voltage reading. However, all three methods only record the radial pressure over a comparatively large region of the die wall.

By using photoelastic techniques however, it seemed that readings which approximated more to point values could be obtained. We found that powders could be compressed in small perspex dies between $\frac{1}{2}$ inch diameter duralumin punches in an ordinary bench vice and up to about 15 fringes could be obtained in the Perspex before cracking took place.

In conventional photoelastic measurement, an araldite model of the system to be analysed is made. Araldite is a very sensitive material, in that stresses of a few tens of pounds per square inch produce several interference fringes. This would have been useless for the present purpose, because although a model die could have been made, no model powder could. Powder behaviour is a non-linear function of the applied stress: the behaviour at 10 lb/in^2 stress is qualitatively unlike that at 10,000 lb/in^2.

Experimental

It has proved possible to compress a tablet in a perspex die, to remove it by means of a duralumin ejector, and to examine its properties. Ejection is difficult, though not impossible, because the strain in a perspex die is much larger than that in a steel die. Usually some degree of lamination or capping of the tablet takes place, indicative of a high degree of compression. Compaction of powders was carried out using a hydraulic

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ram up to a pressure of 60 lb/in^2 (equivalent to 12·7 tons/in^2 on the compact).

The general arrangement of the apparatus is shown in Fig. 1. The Perspex die is mounted between the solid pillar-like extensions fixed to the upper and lower platens of a Tangye hydraulic press, capable of exerting a force of 100 tons. The lower pillar sits on a steel bridge, through which the optical bench passes. The gauge recording pressure in the hydraulic system is fitted with a flexible connecting tube so that it can be positioned on the right of the die. To the left of the die, a micrometer dial gauge, mounted on a magnetic holder, is used to record the lower punch movement during the tabletting operation. The optical bench, 6 feet long, carries a tungsten filament photoflood lamp operated from the mains through a variable transformer and underrun at about 140 V. Light from this passes through a 9 inch diameter condenser lens, through the polariser and the Perspex die, and then on through the analyser to a ciné camera. Stray light is eliminated by a black cloth draped over the press, and by a large internally-blackened cardboard tube covering the distance between the press and the camera. To make the dial and pressure gauge readings visible, it was necessary to provide front lighting by means of small spot-lights.

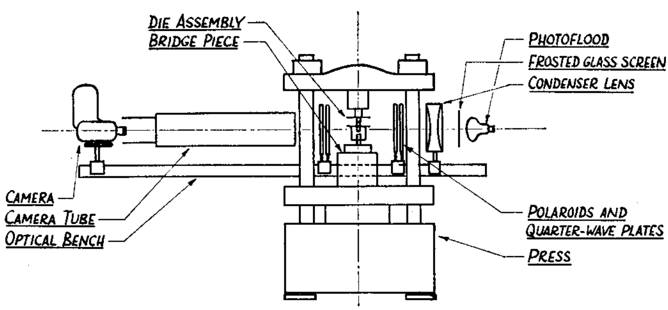

FIG. 1. General arrangement of the apparatus. Compression is applied to the lower punch of the punch and die assembly by the lower platen of the hydraulic press. The optical bench carries the light source, polaroids and camera and keeps them in accurate alignment. The bridge piece carries the force from the lower platen to the punch, whilst preventing the movement being transmitted to the optical bench.

The arrangement of the die is shown in Fig. 2. The die is made from a cylinder of Perspex 2 in long and 2¼ in in diameter, with two flat surfaces on it to give a clear view of the central ½ in diameter hole. A graticule of lines is ruled on one of the flat surfaces to provide reference zeros for fringe measurements. The upper and lower punches are made of free cutting stainless steel, and are a sliding fit in the central hole of the die. A duralumin disc fits around the top punch and rests on top of the Perspex die block. Spacers are sometimes required. Between the disc and a similar one attached to the upper pillar, two small pieces of Perspex are placed; these are so shaped that a vertical force applied to them causes bending in the shank. The shank is sufficiently long to give the characteristic striped pattern of pure bending fringes over a sufficient length to enable the force to be determined by fringe counting. Compressive force is applied only to the lower punch. Radial stresses in the die give a fringe pattern directly. Any tendency of the die to move upwards due to the imposition of shear forces on the die wall by the moving powder is transmitted through the duralumin disc and appears as a fringe pattern in the small Perspex pieces. Two small triangular prisms are placed one above and one below the die, so that an additional view of the fringe pattern inside the die is obtained on a vertical line.

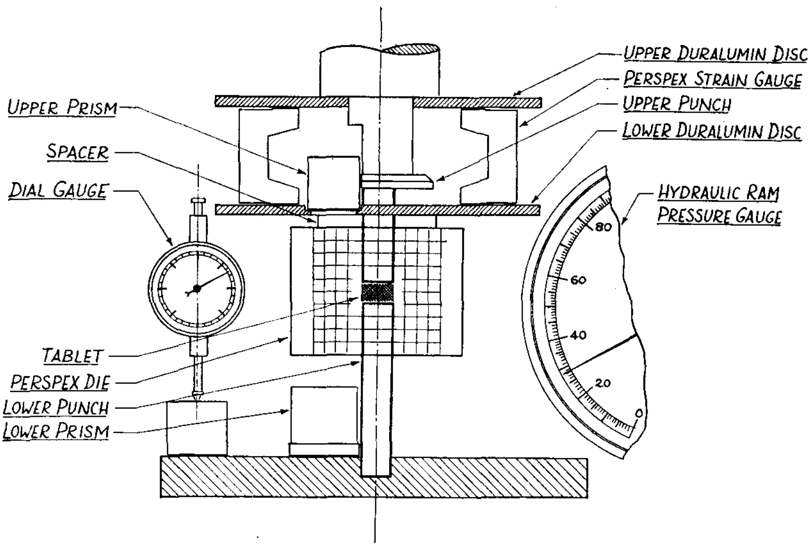

FIG. 2. The punch and die assembly. Radial stress produces fringes in the body of the die. These are viewed directly, and are also inspected along a vertical line by means of the upper and lower prisms which form a periscope. Shear stress at the wall causes upward movement of the die. This is transmitted to the upper Perspex strain gauges, which bend and give fringes in their vertical shanks. Lower punch movement is measured by the small dial gauge, and the force applied to the lower punch is given by the pressure gauge.

A known weight of the powder under study is put into the die, which is placed in position in the press. The lighting and focusing of the camera are adjusted, and the screening for the exclusion of stray light is placed in position. The camera is started, and the press pressure is increased by manual operation of a spring-loaded control lever which operates the valve leading hydraulic fluid to the ram. The pressure is increased at a slow uniform rate; by means of a mirror it is possible to see the pressure and dial gauge faces during operation of the press. An average pressing run takes only a few minutes, and usually occupies about 75 ft of 16 mm Kodachrome 2 ciné film. In the results reported, at the normal speed of 16 frames/sec, the exposure time was 1/40 sec at f.11.

PHOTOELASTICITY THEORY

The basis of photoelasticity is that when a beam of plane-polarised light passes through a piece of transparent plastic material under stress, the beam is split into two components. One of these components is retarded relative to the other, due to its travelling at a different velocity. At the analyser, the two beams recombine, to produce interference fringes which can be photographed. The relative retardation is governed by the properies of the plastic material (usually expressed as a "fringe constant" C), its thickness, d, measured along the light path, and the difference between the two principal stresses acting perpendicularly to the light path.

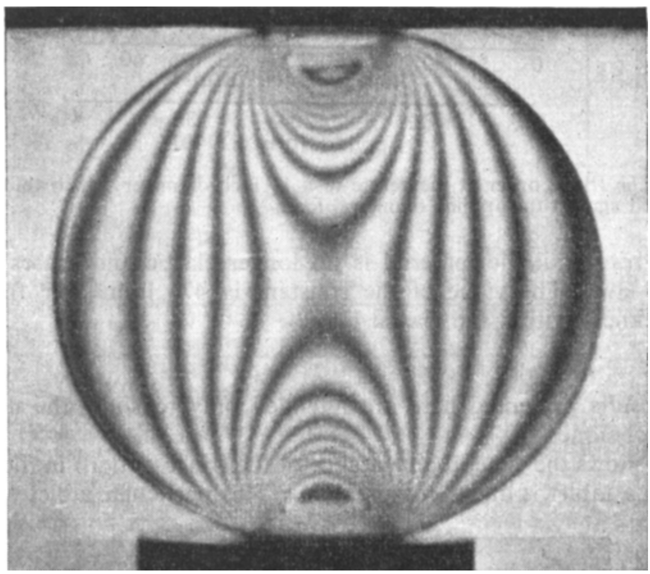

FIG. 3. The fringe pattern obtained when a Perspex disc is loaded along a diameter. For an applied load P, the stress difference at the centre of the disc is $8P/\pi dt$. This enables the fringe constant of the material of the disc to be determined.

Thus

relative retardation $= C \times d \times$ (principal stress difference).

It can be shown (Jessop & Harris, 1949) that the relative retardation will be such as to cause extinction of a beam of light of a particular wavelength whenever the relative retardation is an integral number of wavelengths: this gives rise, in white light, to coloured fringes called "isochromatics". There will also be extinction wherever the directions of the principal stresses are parallel to the polariser and analyser respectively. Such dark lines are called "isoclinics".

MEASUREMENT OF THE FRINGE CONSTANT

If a disc of the photoelastic material is compressed along a diameter, a fringe pattern is formed as in Fig. 3. The principal stress difference at the centre is known to be $8P/\pi dt$ where P is the applied load, d is the diameter and t the thickness of the disc. Fig. 4 is a plot of fringe order at the centre of the disc against the applied force. The plot is linear over most of its length, indicating that the fringe order is proportional

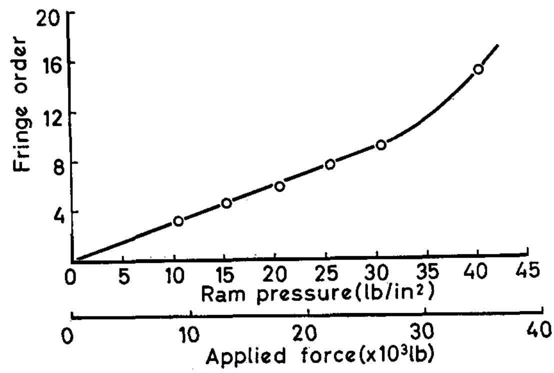

FIG. 4. The fringe order at the centre of a 1½ inch diameter ¼ inch thick Perspex disc plotted against the applied diametral load.

to the stress until the material is no longer elastic and ceases to obey Hooke's law. The value of the constant is 476 lb/in² per fringe per inch thickness within the elastic limit.

Results

The results presented here are intended only to show the usefulness of the technique.

Fig. 5 shows the general appearance of the fringe pattern in the perspex die when a tablet is under compression. This particular tablet was made

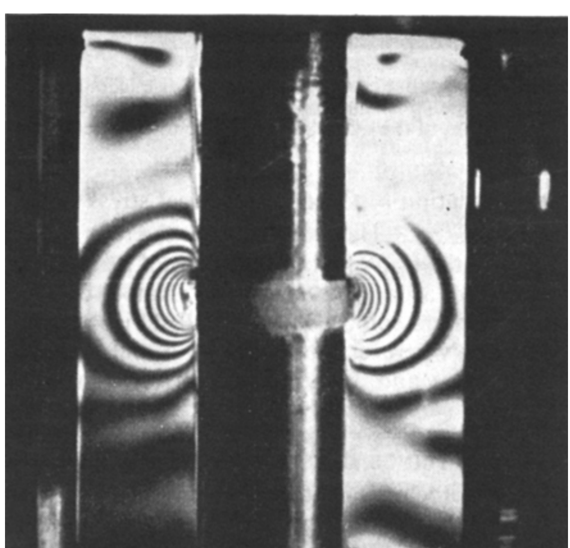

FIG. 5. The appearance of the fringes obtained in the Perspex die when a tablet is being compressed (20–30 mesh aspirin; ½ inch diameter tablet with 7800 lb applied force, equivalent to 39,600 lb/in²)

of 20–30 mesh aspirin, and weighed 1 g. A series of pictures taken as the tablet was compressed enabled readings to be abstracted of the ram pressure and the movement of the lower punch (and thus the extent to which the tablet was reducing in thickness as pressure was applied). The fringe order at this tablet edge was counted and enabled the plot in Fig. 6 to be made.

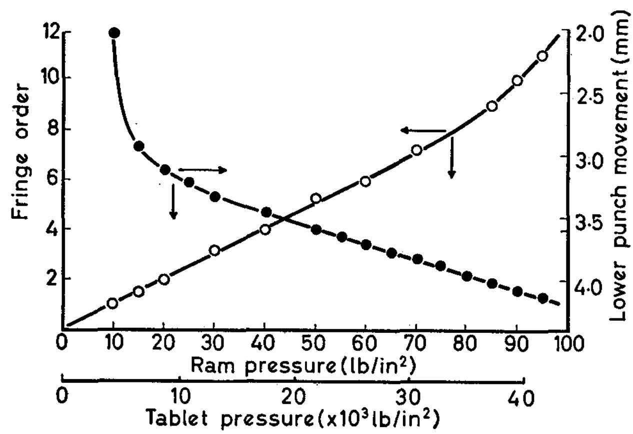

FIG. 6. Fringe order in the die wall immediately adjacent to a tablet (a measure of the radial pressure) plotted against pressure applied. The tablet is that shown in Fig. 5.

The fringe order increased as the punch pressure, and therefore the radial pressure exerted on the die-wall, increased. To assess the constant of proportionality a known radial pressure was applied to the die-wall by applying the punch pressure to a liquid confined in the tablet space. Since the pressure distribution must then be hydrostatic, the radial die-wall pressure must be equal to the applied punch pressure. The difficulty of confining a liquid in the die at the high pressure required was surmounted by wrapping a small amount of silicone putty in a small sack of chamois leather, wired at the neck. With an upper punch recessed in the centre to accept the wired neck of the bag, this was compressed up to a ram pressure of 30 lb/in^2. The fringe pattern was similar to that obtained on compression of a powder, but the fringe order was approximately three times as great for the same applied pressure, over the restricted range which could be covered before leakage occurred. Thus the radial pressure exerted by the tablet was about one-third of the applied pressure. This agrees with the finding of Nelson (1955) that in the compression of unlubricated sulphadiazole the radial die-wall pressure is about 30% of the applied punch pressure.

References

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