International Journal of Pharmaceutics, 3 (1979) 221–230

INSTRUMENTATION AND CALIBRATION OF A SINGLE-PUNCH PRESS FOR MEASURING THE RADIAL FORCE DURING TABLETING

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(Received October 24th, 1978)
(Revised version received January 17th, 1979)
(Accepted February 28th, 1979)

SUMMARY

A single-punch tablet press was instrumented for radial force measurement during tableting by cutting out segments from the die, leaving 5.0 mm of die wall, and by applying strain gauges to these thinner parts. Active horizontal gauges were connected to temperature-compensating vertical gauges to form a Wheatstone bridge with two fixed resistors in the bridge amplifiers. The instrumentation was dynamically calibrated at tableting speed (30 rpm) with rubber-like materials lubricated with molybdenum disulphide (Molykote). The die wall signals were reproducible and increased linearly with the applied pressure. The signals increased with increasing height of the compact. The position of the lower punch had a marked influence on the response. When the lower punch was in a position 10.2 mm below the upper surface of the die, the signal at constant pressure was proportional to the cross-sectional area of the compact. At this position the die wall pressure could be calculated from the regression line of signal/cross-sectional area vs applied pressure. This regression was the same for all tested rubber materials and the estimated relative standard deviation of single determinations at 100 MPa was about 3%.

INTRODUCTION

During one-sided compaction, e.g. tableting in a single-punch press, the applied forces are transmitted axially and radially. Measurement of the radial forces was used as long ago as in 1949 in studies on the compaction process and on the pressure distribution through metal compacts (Duwez and Zwell, 1949; Shank and Wulff, 1949). In the pharmaceutical area, the radial force has been used for calculating, for example, the residual radial force after the compaction, to predict the capping tendency (Obiorah and Shotton, 1976). Plots of radial vs axial pressures were proposed by Long (1960, 1963) to characterize material properties (Summers et al., 1976). The kinetic friction coefficient at the die wall during ejection of metal compacts has been studied in attempts to correlate the coefficients with die abrasion (Mallender and Coleman, 1975; Bockstiegel and Svensson, 1971).

The various instrumentations for radial force measurements have recently been reviewed by Marshall (1972) and Sixsmith (1977). The measurements may either be direct, using split-dies or pistons in contact with the compact, or indirect, by measuring the elastic expansion of the die wall. The latter principle is more commonly used and interferes less with the compact formation. It requires calibration to convert the expansion measurements into force values since the die material, the wall thickness, the dimensions of the compact and its position in the die will influence the results. The choice of equipment depends on the type of study it is intended for and what can be used in the available tablet press. When we wanted to equip our reciprocating tablet press (Hölzer and Sjögren, 1977) for accurate measurements of the radial forces at normal tableting speed we chose to use strain gauges bonded to cut-away segments of the die wall. This device is close to the force origin and can be used without alteration of the press. The aim of this study was to investigate the calibration procedure, the influence of the compact height and the position of the compact in the die.

EXPERIMENTAL

Equipment

A single-punch press, equipped with piezoelectric load washers (Kistler 902 A, linearity ±0.4%, accuracy ±1%) for registration of upper and lower punch forces, was used (Hölzer and Sjögren, 1977). The charge amplifiers (Kistler 5001) had a bandwidth of 180 kHz, – 3 dB. Inductive displacement transducers (Philips PR 9314/10 and 20) were used to determine the positions of the upper and lower punches and to measure the distance between the two punches. The height of the compact was calculated from this difference

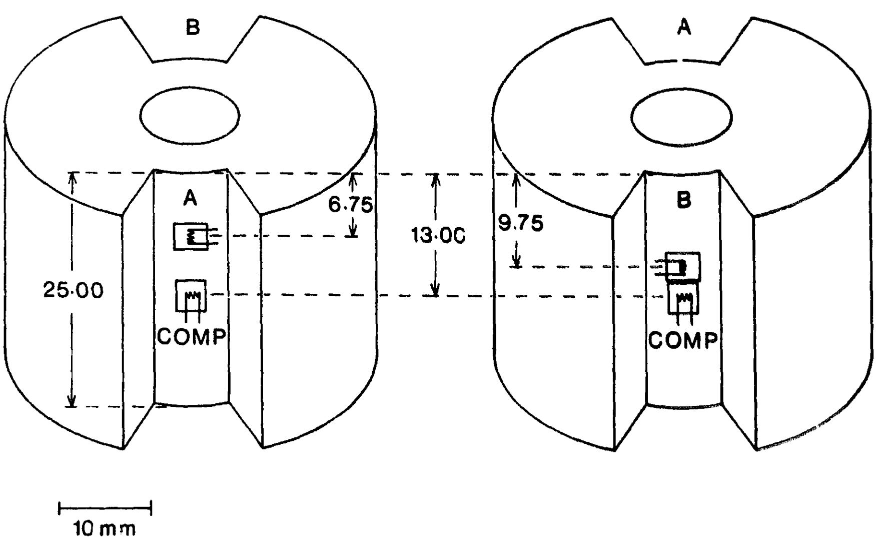

Fig. 1. Diagram of the 11.3 mm die with two cut-away segments showing the positions of bridges A and B.

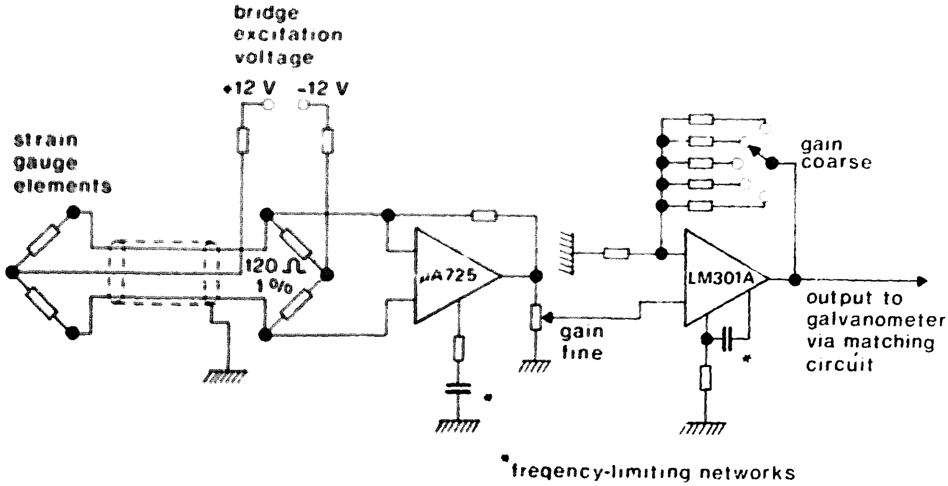

Fig. 2. Block diagram of strain gauge bridge amplifier.

after correction for the elastic deformation of machine parts and punches as previously described (Hölzer and Sjögren, 1978). The elastic deformation was 5.5 μm/kN and was linear within the force range studied. The displacement converters (Philips PR 9871) work with a carrier frequency of 5 kHz and have a bandwidth of 200 Hz, – 3 dB. The signals were recorded with 500 Hz mirror galvanometers on photographic paper in a UV-oscillograph (Ultralette 5656, ABEM, Sweden). The charge amplifiers and galvanometers were calibrated to ±1% as before (Hölzer and Sjögren, 1977).

An 11.3 mm diameter die of 23-12 steel was hardened to 60 HRC after two opposite segments had been cut away (AB Ellips, Johanneshov, Sweden), leaving a die wall thickness of 5.0 mm. The elastic expansion of the thinner wall was used for force registration. Both segments were equipped with an active and a compensating strain gauge (Fig. 1). The phenol/epoxy-backed foil strain gauges (Kyowa, KFC-03-C1-11, 120.0 ± 0.3 Ω, gauge factor 2.10 ± 1.5%, element size 0.3 X 1.4 mm) were bonded to the die wall with cyanoacrylate adhesive (Kyowa CC-15A). The transverse sensitivity perpendicular to the gauge is 3% of the sensitivity in the direction of the principal axis. The active gauges were placed 6.75 and 9.75 mm from the die surface (sides A and B respectively) and the compensating gauges 13.00 mm from the die surface. The active guage and the compensating gauge were connected to make a half-Wheatstone bridge to improve the temperature stability. The bridges were completed with two fixed precision resistors in the two amplifiers (see Fig. 2), and they are referred to below as bridge A and bridge B. The top geometry of the die was restored with silicone bonding cement and the gauges were moisture-proofed with synthetic resin spray. The radial force signals (DWS) are expressed as cm deflection of the galvanometer.

Materials

Breon Polyblend 503 (BP Chemicals, U.K.) is a blend of 52% polyvinyl chloride and 48% nitrile rubber and is available in granule form, size 0.2–1.0 mm.

Nitrile rubber (Gislaveds Gummifabrik AB, Sweden) with 5% zinc stearate, granule form, size 0.1–1.0 mm.

Red rubber powder, size ~0.2 mm, from grinding a rubber stopper.

Black rubber powder, size ~0.1 mm, from grinding car tyres (Gislaveds Gummifabrik AB, Sweden).

Silicone stoppers, diameter 11.2 mm, obtained by molding silicone rubber solution with a hardener (Silastic 382, Dow Corning Corp., Midland, Mich., U.S.A.).

Procedure

The rubber-like materials were lubricated with molybdenum disulphide (Molykote, Kistler) to reduce friction and to prevent vulcanization of the rubber powders. The absence of significant friction could be checked by measuring the difference between the peak punch forces. The die was hand-filled and the tablet machine started at normal speed, 30 rpm. The upper and lower punch forces, the DWS from bridge A and bridge B and the upper and lower punch displacement signals were recorded simultaneously at a paper speed of 50 cm/s.

RESULTS AND DISCUSSION

The calibration is based on the assumption that the rubber-like materials behave like a fluid and that the pressures in all directions are equal, i.e. the upper punch pressure (UPP), the die wall pressure (DWP) and the lower punch pressure (LPP) are equal (Windheuser et al., 1963). Lubrication with molybdenum disulphide was very efficient and the force differences between the punch forces were negligible, less than 1% of maximum load. Only tracings with negligible remaining radial signals after the compression cycle were chosen for calibration calculations. Preliminary trials showed that the maximum radial signals separated about 5 ms relative to the maximum punch signals and so did the upper punch displacement signals. This could partly be explained by differences in the amplifiers. The charge amplifiers have a bandwidth of 180 kHz whereas the bridge-amplifiers' width and the displacement converters' width was limited to 200 Hz, – 3 dB.

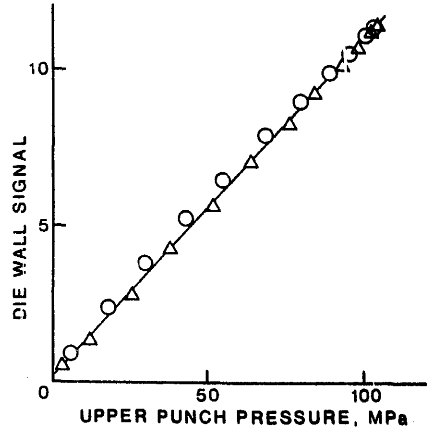

Fig. 3. Die wall signal during compression cycle for lubricated Polyblend 503 material. Lower punch position 10.2 mm, compact height 5.0 mm, bridge B. △, increasing pressure; ○, decreasing pressure.

By changing capacitors in the frequency-limiting networks (Fig. 2), the bandwidth of the bridge-amplifiers could be increased to 500 Hz, – 3 dB, at the cost of a somewhat higher noise. The displacement converters were not changed, as the delay of the displacement signals does not have a great influence on the results and can easily be corrected for when measuring the tracings. Capacitors of 0.001 μF were used in the two frequency-limiting networks of the bridge amplifiers and gave acceptable time delay and noise.

The DWS increased linearly with punch pressure for all calibration materials. The DWS were regressed on UPP with the assumption that the error in UPP is negligible ($<1\%$), and the least square regression technique was used. As a measure of linearity we chose the relative standard deviation of the slope from the linear regression analysis of DWS vs UPP and it was generally 0.5–1.0%. In Fig. 3 an example of a calibration is shown, where the relative standard deviation of the slope was 0.7% and the correlation coefficient 1.00. The Polyblend 503 material gave only a slight hysteresis between up and down signals, in contrast to the other materials, which gave obvious hysteresis. Polyblend 503 was easy to use and was used in the following studies, unless otherwise stated.

Test runs were made using different compact heights at 7 different positions of the lower punch to investigate the influence of these variables on the response from the two bridges. Table 1 gives the results from stepwise multiple linear regression analysis of the data, based on a linear model of the following type:

$$DWS = a + b \cdot UPP + c \cdot HT + d \cdot POS$$

where DWS is the die wall signal, UPP the pressure, HT the compact height, POS the lower punch position and a, b, c and d are constants. Bridge B was less affected by the lower punch position than bridge A.

In another test series the compact height was kept constant at 4.15 ± 0.06 mm (mean ± standard deviation) at 7 lower punch positions. Fig. 4 shows the DWS (mean of 3–5 compacts) at 100 MPa UPP and the DWS reached a maximum at lower punch positions about 7 and 8 mm below the die surface for bridge A and B, respectively. The dependence of the DWS on the position of the compact can be explained by the different distances between the force origin and the active and the compensating gauge and this is

TABLE 1

STEPWISE MULTIPLE LINEAR REGRESSION ANALYSIS OF DIE WALL RESPONSE

Lower punch positions 6.2–12.2 mm below die surface, compact heights 1.6–6.9 mm, pressures 1.0–160.0 MPa. Polyblend 503 with molybdenum disulphide. Number of observations 647.

Variable	% of response explained by the regression	
	Bridge A	Bridge B
Axial pressure	59.2	61.9
Compact height	14.2	16.6
Lower punch position	9.0	3.6

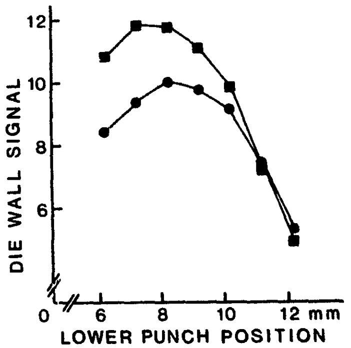

Fig. 4. Die wall signals at 100 MPa upper punch pressure for various lower punch positions and at constant compact height (4.15 ± 0.06 mm) for lubricated Polyblend 503 material. ■, bridge A; ●, bridge B.

probably also the reason for the difference between the two bridges. Both gauges were placed on the cylindrical part of the cut-away segment (Fig. 1). The die-material expands horizontally but to some extent also in the vertical direction resulting in a change of the resistance of not only the active gauge but also of the compensating gauge. As the ampli-

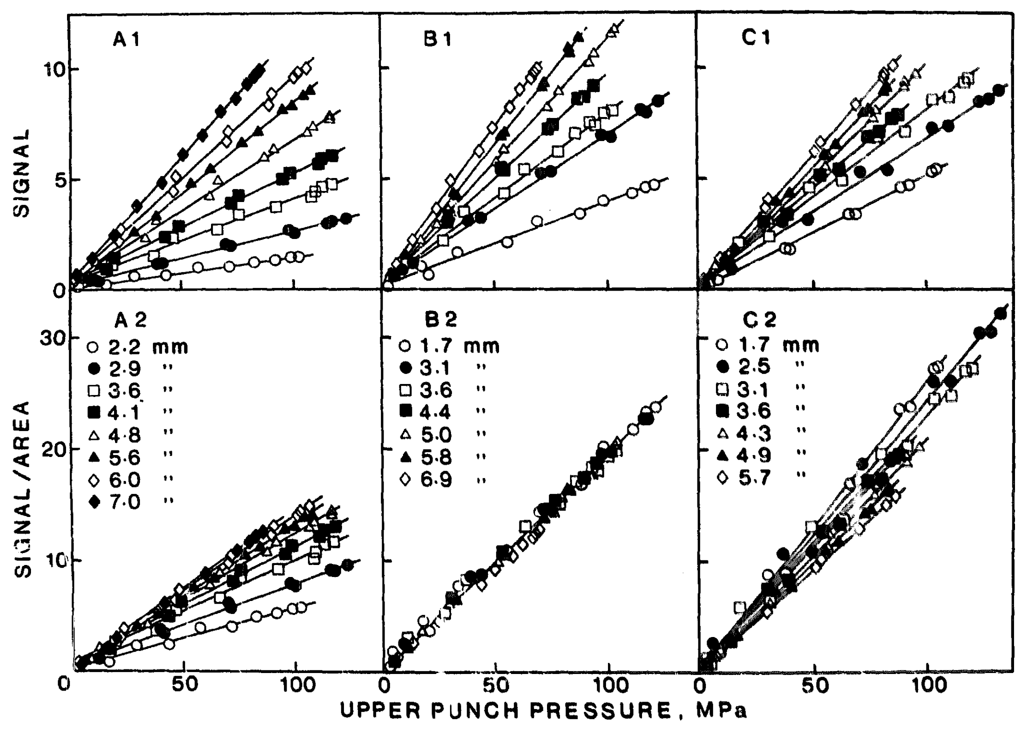

Fig. 5. Influence of compact height on die wall signal (SIGNAL) and on SIGNAL per cross-sectional area (SIGNAL/AREA) at various pressures. Lower punch positions: A, 12.2 mm; B, 10.2 mm; C, 8.2 mm.

tude of the DWS depends on the difference between the resistances of the gauges the DWS will increase when the force origin is removed from the compensating gauge and then gradually decrease when the force origin moves above the active gauge. The greater distance between the gauges of bridge A than of bridge B can explain the higher DWS for bridge A. Regression analysis of DWS vs UPP showed that bridge A gave larger relative standard deviation of the slope than bridge B, e.g. 1.37% and 0.84%, respectively, for the 10.2 mm position.

The DWS increased linearly with pressure at all punch positions shown in Fig. 4. As expected higher compacts gave higher DWS, see Fig. 5. Attempts to compensate for the height variation by dividing by the cross-sectional area of the compact worked only when the lower punch was positioned 10.2 mm below the opening. At lower positions the area compensations were insufficient and at higher positions they were too great, as shown for bridge B in Fig. 5. The same applied for bridge A, which is surprising considering the different positions of the strain gauges. At the lower punch position 10.2 mm the DWS/AREA was independent of the compact height which means that the DWS were proportional to the radial *force* and that the radial *pressure* of a compact of any height within the limits tested could be calculated by dividing the DWS by the cross-sectional area and by using the relationship shown in Fig. 5B2.

The reproducibility of the calibration was tested on 6 different occasions over a period of 6 months using bridge B at the depth of 10.2 mm and lubricated Polyblend 503. The compact heights were kept constant at 4.00 ± 0.10 mm (mean ± standard deviation). The relative standard deviation of the slope of DWS/AREA vs UPP did not exceed 2%. Sixteen

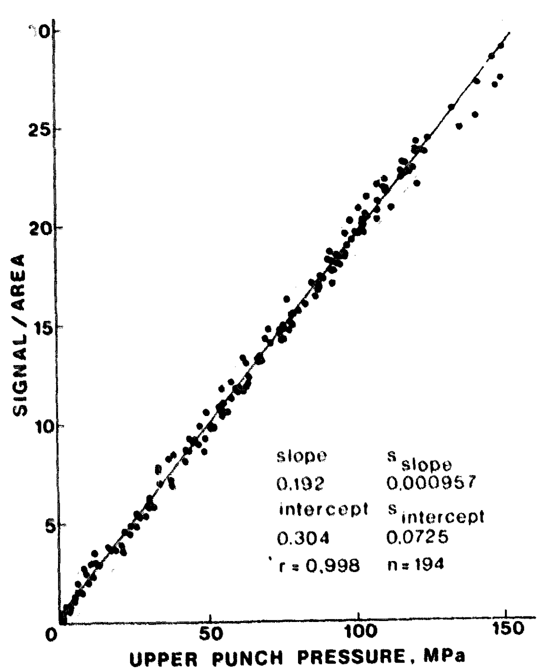

Fig. 6. Calibration regression of die wall signal per cross-sectional area (SIGNAL/AREA) for lubricated Polyblend 503 material at a lower punch position of 10.2 mm and at various compact heights. The 95% confidence limits of single predicted values are indicated with dotted lines.

determinations of DWS/AREA, calculated from the regression lines, gave a mean value of 19.72 ± 0.47 at 100 MPa UPP. Similar calculations on results from two experiments with each of the 5 materials gave 19.68 ± 0.43. Thus all materials tested gave similar results and the calibration is consequently valid for different materials. The reproducibility was good, even when different compact heights were used, as can be seen from Fig. 6, which gives results from 15 experiments with lubricated Polyblend 503 compacts with heights varying from 1.7 to 6.1 mm. The regression equation in Fig. 6 is

$$\text{DWS/AREA} = 0.304 + 0.192 \cdot \text{UPP}$$

According to Sokal and Rohlf (1969), the standard deviation is ($s_{\hat{y}}$) of *one* predicted value Y_i (DWS/AREA) for a given X_i (UPP) is:

$$s_{\hat{y}} = \left[s^2{}_{yx}\left(1 + \frac{1}{n} + \frac{(X_i - \bar{X})^2}{\sum x^2}\right)\right]^{1/2}$$

where $s^2{}_{yx}$ is the unexplained mean square deviation from regression of Y on X, n (= 194) is the sample size of the calibration regression, $\bar{X}$ (= 63.101) the mean pressure values of the calibration curve and Σx^2 (= $3.43 \cdot 10^5$) the sum of squares of the deviations from the mean. At an X_i of 100 MPa the DWS/AREA (Y_i) is 19.50, with a standard deviation ($s_{\hat{y}}$) of 0.56, which is in very good agreement with the results calculated above. Fig. 6 also gives the 95% confidence limits of the predicted Y values and it is obvious that the accuracy was acceptable over a wide pressure range. The relative standard deviation of the predicted DWS/AREA value was about 3% of 100 MPA, which is in agreement with the errors stated by Long (1960), Jungersen and Jensen (1970) and Lindberg (1972), but it was considerably higher at lower pressures. The regression of DWS/AREA on UPP can be used for calculation of DWP from known DWS/AREA values by rearranging

$$\text{DWP} = \text{UPP} = (\text{DWS/AREA} - 0.304)/0.192$$

If we have a DWS/AREA of 20.00 and wish to calculate the corresponding DWP, the equation above gives a value of 102.58 MPa. It is not appropriate to assign the above-mentioned standard deviation ($s_{\hat{y}}$) to this value, but confidence limits can be calculated according to Sokal and Rohlf (1969), given 102.54 ± 5.76 MPa (radial pressure ±95% confidence limits), see Appendix.

APPENDIX

Calculation of DWP from DWS/AREA

Given a DWS/AREA reading of 20.00 for a radial signal, what is the radial pressure, assuming that the experimental setup was identical to that used during the calibration?

Since

$$\text{DWS/AREA} = 0.304 + 0.192 \cdot \text{UPP}$$

$$DWP = UPP = \frac{DWS/AREA - 0.304}{0.192} = \frac{20.00 - 0.304}{0.192} = 102.58$$

we define a quantity D as:

$$D = b^2_{yx} - t^2_{0.05(n-2)} \cdot s^2_b$$

where b_{yx} is the slope of the regression of DWS/AREA on UPP, $t_{0.05}$ the Student t value at $P = 0.05$ and at n – 2 degrees of freedom, n is the sample size of the calibration, and s_b the standard deviation of the slope.

$$D = 0.192^2 - 1.97^2 \cdot 0.000957^2$$
$$D = 0.0369$$

We then define another quantity H as:

$$H = \frac{t_{0.05(n-2)}}{D} \cdot \left[s^2_{yx}\left[D\left(1 + \frac{1}{n}\right) + \frac{(Y_i - \bar{Y})^2}{\sum x^2}\right]\right]^{1/2}$$

where Y_i is the predicted DWS/AREA reading and $\bar{Y}$ the mean DWS/AREA reading of the regression in Fig. 5. The other variables are defined as above and as defined in the calculation of the standard deviation ($s_{\hat{y}}$) of *one* predicted DWS/AREA value.

$$H = \frac{1.97}{0.0369}\left\{0.312\left[0.0369\left(1 + \frac{1}{194}\right) + \frac{(20.00 - 12.42)^2}{3.43 \cdot 10^5}\right]\right\}^{1/2}$$

$$H = 5.76$$

The 95% confidence limits are

$$L = \bar{X} + \frac{b_{yx}(Y_i - \bar{Y})}{D} \pm H$$

$$L = \left[63.10 + \frac{0.192(20.00 - 12.42)}{0.0369}\right] \pm 5.76$$

$$L = 102.54 \pm 5.76$$

Note that the limits are symmetrical arround 102.54 and not around the predicted DWP value of 102.58.

ACKNOWLEDGEMENTS

The authors are grateful to Mr. Ulf T. Jonsson for help with the construction of the electronic equipment, to Mr. Elof Gustafsson for bonding the strain gauges, and to Gislaveds Gummifabrik AB for supplying the rubber samples.

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