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Simultaneous monitoring of tamping force and pin displacement (F-D) on an Hofliger Karg capsule filling machine

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Summary

Linear variable displacement transducers (LVDTs) were added to a previously instrumented Hofliger-Karg GKF 330 capsule filling machine. The new instrumentation provided displacement measurement in addition to force measurement. Greater force and displacement were encountered when using the 2.0 mm overload spring versus the 1.6 mm overload spring. Plug weight did not increase dramatically due to a high degree of elastic recovery post compression resulting in little comparative advantage of the 2.0 mm overload spring in terms of reaching the target weight. The force-displacement curve generated uniquely represents the compaction process of this filling machine. Area-under-the-curve calculations of the force-displacement curve to determine work of compaction confirm that as expected a much smaller amount of work is done in capsule plug compaction compared to tableting.

Introduction

Instrumentation of tablet and capsule filling machines may be approached using one of any number of different techniques (Marshall, 1983). The application of instrumentation to automatic capsule filling machines has proved to be valuable in evaluating capsule formulation and manufacture (Cole and May, 1975; Shah et al., 1983, 1986, 1987). Automatic capsule filling machines whose operating principles resemble tableting to the extent that powder plugs formed by tamping (i.e., compressed) are ejected into the capsule shells did not begin to appear until the 1960s. It wasn't until 1975 that the application of instrumentation to such machines was first reported by Cole and May (1975) who installed a strain gauged tamping piston in a Zanasi LZ-64 dosator machine. Later, Mehta and Augsburger (1980) made simultaneous measurement of force and displacement on a Zanasi LZ-64 machine. More recently, Shah et al. (1983) reported the first application of instrumentation techniques to an Hofliger-Karg (H & K) GKF 330 dosing disk machine. Unlike the simpler dosator machines, which form plugs through a single tamping stroke, dosing disk machines build up plugs progressively through a multiple tamping process. To obtain tamping force measurements in

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the dosing disk machine, Shah et al. (1983) installed tamping pins fitted with strain gauges at different tamping stations. Studies with this system provided new insights into the plug formation process in such machines and its effect on drug dissolution (Shah et al., 1986, 1987). However, the multiple tamp process is complex, and a full characterization and understanding of plug formation was hampered by the inability to monitor actual tamping pin movement along with tamping pin force. Thus, the present study describes the development of instrumentation to determine displacement on an H & K GKF 330 machine previously instrumented to measure tamping force (Shah et al., 1983). Information pertaining to displacement of the tamping pin from the dosing disk during compaction is useful in many areas. For instance, it will now be possible to calculate energy consumption for formation of capsule plugs and make comparisons between different formulations. Programming of a compaction simulator will now be possible. This additional information will make possible the development of a better understanding of low pressure compaction, the mechanism and its ramifications for drug release. The force-displacement measurements reported herein were made while using different types of overload springs which are available for use in the dosing disk machine. Information pertaining to the effects of the different types of springs may be influential in the decision of which spring to use for a particular formulation.

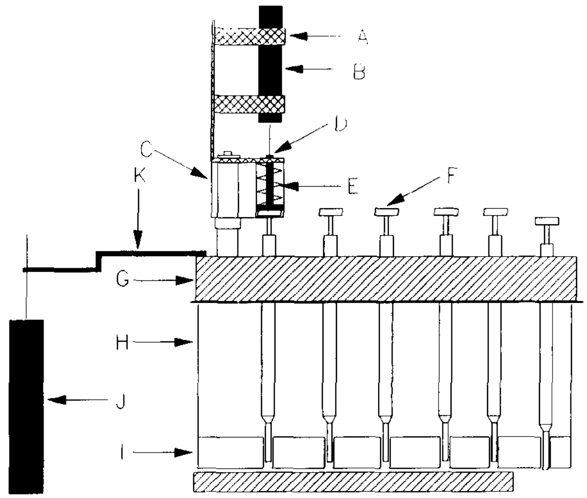

Fig. 1. A, LVDT holder; B, LVDT which measures tamping pin movement; C, tamping pin holder block; D, interface pin; E, overload spring; F, instrumented tamping pin; G, brass ring; H, powder bowl; I, dosing disk; J, LVDT which measures movement of brass ring; K, steel arm.

Materials and Methods

The new instrumentation added to the H & K GKF 330 (Robert Bosch Packaging, Machinery Division, Piscataway, NJ) consisted of two linear variable displacement transducers, LVDTs. Illustrated in Fig. 1 is the instrumented tamping assembly. Photographs of the actual instrumentation are shown in Fig. 2a–c. An LVDT (Model DCT 1000A, RDP Electrosense, Phoenixville, PA) with a working range of ± 25 mm was mounted on the base of the H & K machine. The rod of the LVDT was connected to the brass ring of the H & K via a machined stainless-steel arm (Fig. 2a). This made possible monitoring of the movement of the brass ring upon which the tamping pin holder assemblies are anchored.

A second LVDT (Model 0244, Trans-Tek Inc., Ellington, CT) with a working range of ± 25 mm was mounted on a tamping pin block assembly, situated directly above the center tamping pin (Fig. 2b). The core rod of the LVDT was attached to a modified pin which rests upon the tamping pin head and under the overload spring. This pin serves as the interface between the tamping pin and the overload spring. A new interface pin was designed to extend upward through the overload spring and the cover plate of the tamping pin block assembly. The plate which is secured to the top of the tamping pin holder block assembly was modified by adding three holes, through which the interface pin could extend up towards the LVDT. The threaded end of the LVDT core rod was screwed into the top of the interface pin. The provision of three holes in the plate made possible the connection of the LVDT to any of the three tamping pins; however, in practice, only the central tamping pin was fitted to the LVDT. This second LVDT provided for measurement of overload

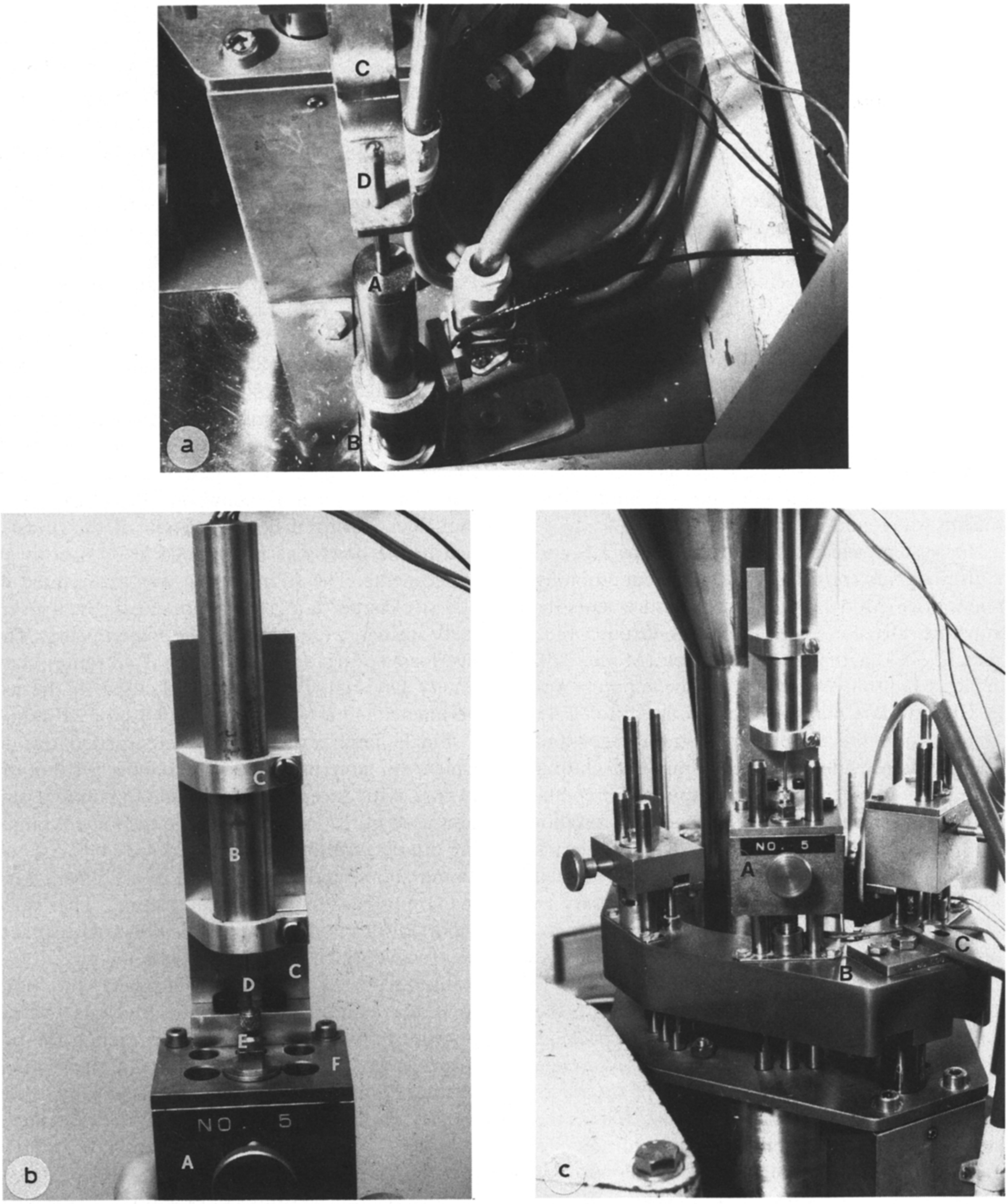

Fig. 2. (a) Top view showing LVDT (A) mounted upon the base plate (B) of the H & K and the stainless-steel linkage (C) of the core rod (D) to the brass ring. (b) Tamping pin holder block (A) upon which LVDT (B) and special support bracket (C) are mounted. The threaded end of the core rod (D) fits into the upper end of the interface pin (E) which extends up from the top of the tamping pin through a modified plate (F). (c) Overall view of tamping assembly showing instrumented tamping pin holder block (A) mounted to the brass ring (B). The linkage to the large LVDT which monitors ring movement also can be seen (C).

spring movement. The combination of ring movement from the first LVDT and overload spring movement from the second LVDT allowed for precise measurement of the actual tamping pin penetration into the dosing disk. The overall assembly is illustrated in Fig. 2c. Until this time, tamping pin penetration could be set only to a theoretical penetration. Other useful parameters which may be determined from the combined information of the two LVDTs include dwell time, rate of tamping pin movement through the powder bed, plateau time, plug length, and powder displacement.

The LVDTs were calibrated by placing the body of the LVDT in a fixed position and attaching the core rod to the movable head of a physical testing machine (Model 1000, Instron Corp., Canton, MA). LVDT voltage output was recorded for various positions of the core rod. A linear relationship was found to exist.

The system which was used to generate and monitor signals consisted of a signal conditioning preamplifier (Model 2310, Micromeasurements Instruments Division, Measurements Group, Inc., Raleigh, NC), a digital oscilloscope (Model 310, Nicolet, Madison, WI), and a microcomputer (Apple IIe, Apple Computer, Inc., Cupertino, CA). Excitation for the tamping pin strain gages and LVDTs was provided by the preamplifier. Output signals were directed to the preamplifier for conditioning before being sent to the digital oscilloscope. The oscilloscope is a two channel device capable of storing only one waveform per channel. Initially, memory limitations made it necessary to use another device for the storage of waveforms. This was a prerequisite for acquiring a large amount of data which could be evaluated at a later time. The microcomputer was linked to the oscilloscope via an RS232 line. A software package (Apple/31), provided with the digital oscilloscope facilitated the storage and evaluation of the waveforms generated on the H & K 330. Later, the Nicolet 310 unit was modified by the addition of a dual 3.5 inch disk drive unit. This allowed for storage of the waveforms in the Nicolet. It was unnecessary to use the Apple IIe computer after this modification of the Nicolet 310. Waveforms stored on the Nicolet 310 disk drive unit were converted into file formats compatible with other software commonly used in this laboratory. The waveforms were smoothed with Asyst software (version 3.0, Asyst Software Technologies, Inc., Rochester, NY). The smoothing resulted in the elimination of random noise found in some waveforms.

Experiments were carried out using two different formulations. The first formulation was microcrystalline cellulose (Avicel PH 101, FMC Corp., Philadelphia, PA) with 0.1% magnesium stearate (Mallinckrodt, Inc., St. Louis, MO). The second formulation consisted of anhydrous lactose (Direct Tableting Grade, Sheffield Products, Norwich, NY) and 0.5% magnesium stearate. 3 kg batches were made by blending the magnesium stearate and excipient in a V-blender for 10 min. Both formulations were filled into size no. 1 gelatin capsules (Capsugel, Greenwood, SC) using a 15.8 mm thick dosing disk. The height of the powder bed in the bowl was maintained at 30 mm in all experiments. The filling speed was maintained at 100 strokes per min. Two different types of overload springs were used in the experiments. The overload springs (Robert Bosch Packaging Machinery Division, Piscataway, NJ) used in the experiments had a wire diameter of 1.6 or 2.0 mm.

Single tamp experiments were carried out in which all tamping pins were removed from the H & K with exception of the single tamping station used in the experiment. The theoretical tamping pin penetration settings used were 0, 5, 10 and 14 mm. Force and displacement measurement were made for each experimental setting. The value reported for pin displacement is the peak displacement value which corresponds with the peak force. Values reported for pin displacement and peak force are a mean of three measurements. Actual penetration was calculated by subtracting the pin displacement from the tamping pin penetration setting. Plug length was calculated by subtracting the actual penetration from the dosing disk thickness. Plug weight determinations are a mean of 10 measurements. Double tamp experiments were performed with various combinations of theoretical tamping pin penetration settings being used for the two stations.

Results and Discussion

Waveforms

Fig. 3a is a movement-time curve for the brass ring. The brass ring moves downward carrying the tamping pins through the powder bed and into the dosing disk. It can be seen from the curve that the movement is continuous until the brass ring reaches its lowest point of travel, at which point the tamping pin holder assemblies are at their lowest position relative to the dosing disk. After a slight pause at this point, the brass ring then begins movement upward. There is a halt in the travel of the brass ring after moving up 2 mm from the lowest point which is reflected in the plateau region in the curve. During the plateau period, the bushings on the other side of the machine which transport the capsules move to the next position. After this pause, the brass ring resumes its upward movement until it reaches the top position, at which point another cycle begins. The force-time curve in Fig. 3b shows an increase in force up to a peak at which time the force decreases until the plateau is reached, after which a rapid decrease in force is seen as decompression resumes. The pin displacement curve, seen in Fig. 3c, increases to a maximum and then decreases to a plateau after which it decreases to zero. An interesting point when both curves are examined is that the displacement curve remains at zero until a minimum yield force is reached. This force is greater for higher wire diameter overload springs. Table 1 lists interesting points which can be determined from the force and displacement curves.

Springs

Compressing the springs using a physical testing machine (Instron Model 1000, Instron Corp., Canton, MA) demonstrates the wide differences in spring tension. The 1.6 mm spring was compressed 12.9 mm by a 100 N force whereas the 2.0 mm spring was compressed only 5.5 mm by a 100 N force. The choice of overload spring to be used in filling a formulation could be a rather critical decision when consideration is given to the far reaching effects of the spring. During the plug formation process the force at which the overload spring begins to compress and thereby decrease the rate and extent of entry of the tamping pin into the dosing disk is dependent upon the strength of the spring. One could expect greater forces to be achieved by the larger wire diameter springs. Of course, this could directly affect plug strength, plug disintegration, and drug dissolution from the plug. In many production facilities, the 2.0 mm wire diameter is often preferred due to its greater durability. The larger wire diameter springs are also favored when working with formulations having low bulk densities and/or high fill weight. This ability to reach higher compression forces is believed to enable the attainment of greater target weights due to greater powder displacement.

Single and double tamp results

The plugs made from the anhydrous lactose formulation were found to reach higher peak forces during the compaction process than the microcrystalline cellulose plugs. This was true over the range of tamping pin penetration settings used in the experiments (Tables 2 and 3). Because of its

TABLE 1

Data which can be obtained from a comparison of force and pin-displacement waveforms

Pin penetration setting (mm)	Peak force (N)	Force at plateau (N)	Pin displacement at peak (mm)	Pin displacement at plateau (mm)	Plug length at peak (mm)	Contact time (ms)	Time to peak force (ms)
0	8.5	0.0	0.5	0.0	15.8	80	54
5	48.5	11.2	2.6	0.7	13.4	206	80
10	107.0	69.3	5.8	3.9	11.6	250	99
14	158.4	118.7	8.6	6.5	10.4	280	117

TABLE 2

Effect of spring type and penetration setting on tamping pin displacement, plug weight, and peak force for anhydrous lactose and 0.5% magnesium stearate in a single tamp experiment at station no. 5

Pin penetration setting (mm)	Pin displacement (S.D.) [a] (mm)	Actual pin penetration (mm)	Plug length (mm)	Plug weight (S.D.) [b] (mg)	Peak force (S.D.) [c] (N)
1.6 mm spring					
0	0.8 (0.046)	−0.8	16.6	330 (0.87)	21.1 (0.39)
5	4.5 (0.054)	0.5	15.3	344 (2.42)	48.9 (0.19)
10	8.2 (0.091)	1.8	14.0	354 (7.96)	76.6 (0.51)
14	11.7 (0.051)	2.3	13.5	357 (16.53)	102.6 (0.00)
2.0 mm spring					
0	0.8 (0.040)	−0.8	16.6	321 (3.24)	17.8 (1.36)
5	3.9 (0.042)	1.1	14.7	351 (3.81)	75.3 (0.39)
10	7.4 (0.016)	2.6	13.2	365 (10.77)	140.2 (0.34)
14	10.7 (0.060)	3.3	12.5	373 (19.70)	200.6 (1.27)

[a] $n = 3$.
[b] $n = 10$.
[c] $n = 3$.
S.D., standard deviation.

lower initial bulk density, microcrystalline cellulose is capable of a higher degree of densification than is the anhydrous lactose. Accordingly, powder displacement was greater for microcrystalline cellulose than for anhydrous lactose. For the same reason higher forces were achieved for the

TABLE 3

Effect of spring type and penetration setting on tamping pin displacement, plug weight, and peak force for microcrystalline cellulose and 0.1% magnesium stearate in a single tamp experiment at station no. 5

Pin penetration setting (mm)	Pin displacement (S.D.) [a] (mm)	Actual pin penetration (mm)	Plug length (mm)	Plug weight (S.D.) [b] (mg)	Peak force (S.D.) [c] (N)
1.6 mm spring					
0	0.0 (0.000)	0.0	15.8	172 (2.99)	14.6 (0.00)
5	2.8 (0.151)	2.2	13.6	187 (4.84)	35.5 (0.70)
10	6.3 (0.080)	3.7	12.1	196 (3.56)	62.8 (0.51)
14	9.3 (0.160)	4.7	11.1	198 (1.51)	84.2 (1.85)
2.0 mm spring					
0	0.3 (0.033)	−0.3	16.1	165 (2.30)	8.3 (0.19)
5	2.3 (0.032)	2.7	13.1	191 (1.87)	45.2 (1.36)
10	5.1 (0.070)	4.9	10.9	201 (3.37)	94.3 (0.78)
14	7.9 (0.072)	6.1	9.7	201 (2.11)	150.2 (1.36)

[a] $n = 3$.
[b] $n = 10$.
[c] $n = 3$.
S.D., standard deviation.

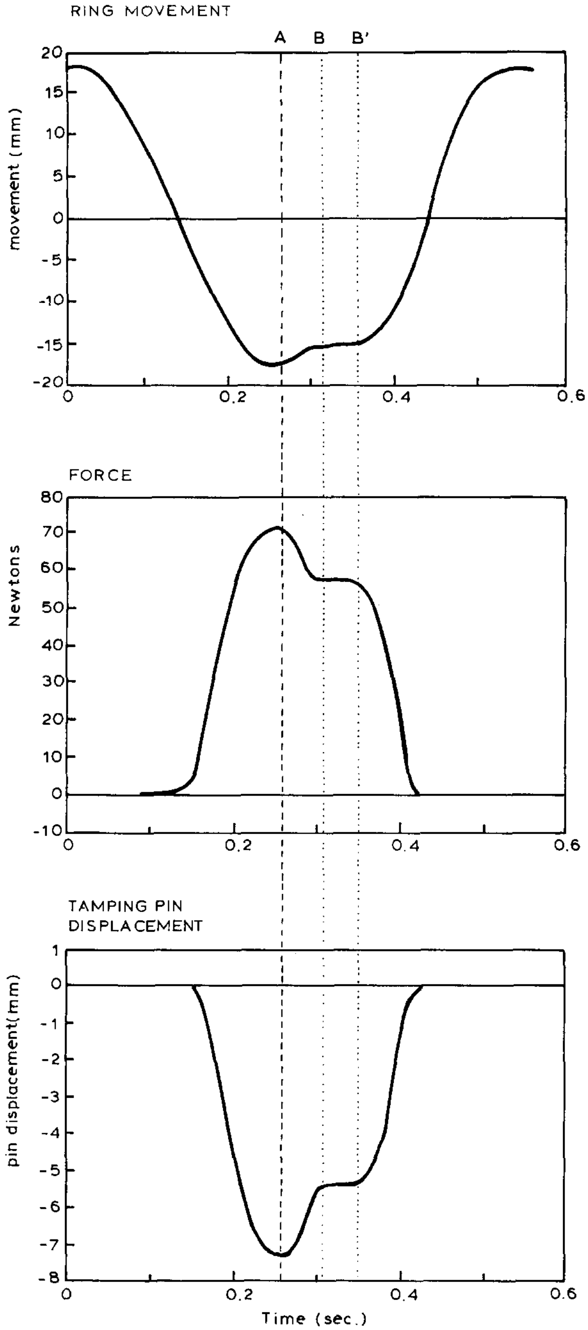

Fig. 3. (a) Movement-time curve for the brass ring. (b) Force-time curve. (c) Pin displacement-time curve.

anhydrous lactose than for the microcrystalline cellulose formulation. Given the same filling conditions, plugs made of anhydrous lactose were longer and weighed more than those made of microcrystalline cellulose. Significant differences in displacement and force against the tamping pin were seen for the various types of overload springs used in the experiment. When the new 2.0 mm wire diameter spring was installed, peak forces were nearly twice the magnitude of those reached when the new 1.6 mm wire diameter spring was installed (Tables 2 and 3). This was true for both of the formulations used in the experiments. In some cases, displacement of the overload spring was seen even when the theoretical tamping pin penetration was set at 0 mm. This phenomenon is reflected in the tables as a net negative actual penetration. One reason for choosing the 2.0 mm wire diameter spring over the 1.6 mm spring is to achieve greater compaction, thus allowing more void volume to remain in the dosing disk cavity post-compression. Since this void volume is filled in with powder from the powder bowl as the machine indexes from one tamping station to the next, providing an adequately free-flowing formulation is used, higher plug weights should be achieved with the larger wire diameter springs. However, it was found in the present study that weights were not markedly affected by the higher forces reached using the larger wire diameter springs. Although weights were found to be higher when using the 2.0 mm wire diameter spring than when using the 1.6 mm spring, the differences were small in relation to the large differences in peak force achieved. This was true for the single tamp study as well as the double tamp study (Table 4). Data obtained in a double tamp study (Table 4) reflect measurements for station 5 after a tamp at station 4. As the penetration setting at station 4 is increased, more powder enters the dosing disk for tamping at station 5. A greater degree of consolidation after the first tamp results in less actual penetration of the tamping pin at station 5. The increase in consolidation, caused by greater tamping pin penetration settings results in increased plug weight and peak force at station 5. Had tamping at three or more consecutive stations been studied, it is possible that the multiplicative effect of a greater number of tamps would have resulted in greater fill weight differences.

A force-displacement curve (Fig. 4) obtained in

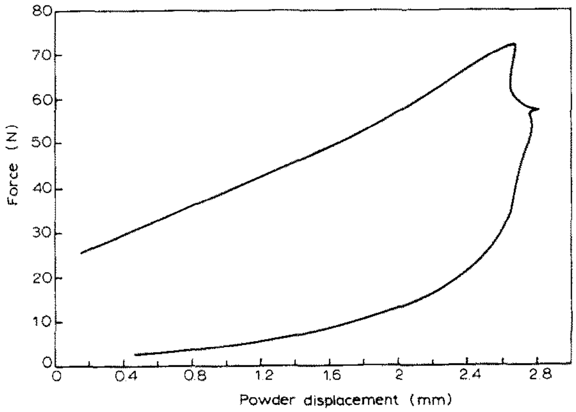

Fig. 4. Force-displacement curve (single tamp experiment; 1.6 mm spring).

a single tamp experiment using microcrystalline cellulose is seen to represent uniquely the tamping process of the H & K. A force is registered before any powder displacement is detected due to resistance against the tamping pin as it travels through the powder bed prior to reaching the dosing disk. This verifies the finding by Shah et al. (1986) who stated that effective compression begins before the tamping pin enters the dosing disk. After reaching a peak force, powder displacement is seen to decrease somewhat until the plateau portion of the curve is reached. At the plateau, additional powder displacement occurs as an almost constant force is maintained on the powders due to the tension in the overload spring. This is seen with the 1.6 mm and 2.0 mm wire diameter overload spring. After the plateau, the curve returns to the baseline exhibiting a high degree of elastic recovery. The elastic recovery is possibly the reason why higher forces seen with the higher wire diameter overload spring do not seem to correspond with greatly increased plug weights.

Calculation of area-under-the-curve (AUC) of the force-displacement curves were made to determine the work of compaction. This method of determining the work of compaction is based upon the work of DeBlaey and Polderman (1971). These authors suggested that in order to determine net work of compaction that the die wall friction be

TABLE 4

Effect of spring type and penetration setting on tamping pin displacement, plug weight, and peak force for microcrystalline cellulose and 0.1% magnesium stearate in a double tamp experiment at stations 4 and 5 (station no. 5 pin penetration was held constant at 10 mm for all measurements)

Pin penetration setting [a] (mm)	Pin displacement [b] (S.D.) [d] (mm)	Actual pin penetration (mm)	Plug length (mm)	Plug weight (S.D.) [e] (mg)	Peak force [c] (S.D.) [f] (N)
1.6 mm spring					
0	7.4 (0.115)	2.6	13.2	203 (2.54)	70.6 (1.72)
5	8.4 (0.166)	1.6	14.2	213 (2.47)	79.3 (0.70)
10	9.0 (0.129)	1.0	14.8	219 (4.16)	83.2 (0.85)
14	9.2 (0.091)	0.8	15.0	225 (2.01)	84.8 (1.01)
2.0 mm spring					
0	6.3 (0.032)	3.7	12.1	204 (7.32)	119.4 (0.34)
5	7.3 (0.080)	2.7	13.1	218 (6.94)	137.7 (0.78)
10	8.0 (0.064)	2.0	13.8	233 (2.67)	151.0 (1.46)
14	8.0 (0.088)	2.0	13.8	241 (4.11)	151.3 (1.21)

[a] Station no. 4.
[b] Station no. 5.
[c] Station no. 5.
[d] $n = 3$.
[e] $n = 10$.
[f] $n = 3$.
S.D., standard deviation.

TABLE 5

Work of compaction for microcrystalline cellulose and 0.1% magnesium stearate and anhydrous lactose and 0.5% magnesium stearate using either 1.6 mm spring or 2.0 mm spring

Pin Penetration setting (nm)	Microcrystalline cellulose		Anhydrous lactose	
	1.6 mm spring network (%R.S.D.) [a] (Nm)	2.0 mm spring network (%R.S.D.) [a] (Nm)	1.6 mm spring network (%R.S.D.) [a] (Nm)	2.0 mm spring network (%R.S.D.) [a] (Nm)
5	0.036 (1.48)	0.070 (6.11)	0.017 (7.13)	0.065 (16.64)
10	0.098 (4.42)	0.22 (1.19)	0.055 (3.24)	0.14 (22.03)
14	0.14 (12.63)	0.40 (3.16)	0.089 (7.59)	0.24 (3.85)

[a] $n = 3$.
%R.S.D., % relative standard deviation.

subtracted from the work done by the upper punch resulting in a term for lower punch work. The work of elastic recovery was subtracted from the lower punch work term in order to determine net work of compaction. The instrumentation on the H & K GKF 330 allows the monitoring of elastic recovery of the powder being compacted by monitoring powder displacement during decompression. This differs from the method used by DeBlaey and Polderman (1971) who subjected the tablet to a second compaction event in order to determine elastic recovery. Using the instrumentation now on the H & K GKF 330 it is not possible to measure force lost to the die wall. Such a measurement would require a major modification to the dosing disk assembly. Nevertheless, the net work of compaction values, seen in Table 5 (determined by subtracting the work of elastic recovery from the work of the tamping pin) are only a small fraction of those reported in tableting research work by DeBlaey and Polderman (1971) which ranged from 0.72 to 4.31 Nm. More recently, Ragnarsson and Sjogren (1985) reported higher net work of compaction values for tablets ranging from 5.80 to 9.99 Nm.

Summary

New instrumentation has been successfully added to an H & K GKF 330 capsule filling machine. This instrumentation required only minimal modification to the machine, and did not interfere with the normal running of the machine. The new LVDTs help to provide a better picture of the compaction process than was available previously by allowing for insight into movement of the brass ring as well as movement of the overload spring. These measurements now permit the correct assessment of tamping pin penetration into the dosing disk. It is clear that the choice of which type of spring to be used in the H & K will have an effect upon the compaction profile of the materials studied. Higher forces were generated while using the 2.0 mm wire diameter spring for both formulations tested. The higher forces may affect bonding of particles, plug strength, porosity, and release rate of active ingredients from the plug.

The addition of LVDTs to the previously installed instrumentation on the H & K makes possible the detailed analysis of the multiple tamping process and of plug compaction in dosing disk machines. Parameters are now available which permit the calculation of energy of compaction for the plugs, as well as the determination of pressure-density relationships. A unique system has been developed which is useful for data collection and evaluation in any instrumentation process.

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