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MASTER

The Friction Index and
Pressing Characteristics of
Spherical Stainless Steel
Powder and Oxidized Copper Powder

UNITED STATES
ATOMIC ENERGY COMMISSION
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The Friction Index and
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by

H. Sheinberg

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IN REPLY
REFER TO:

January 23, 1968

TO: Holders of LA-3739
SUBJECT: ERRATA TO LA-3739

Page 4, Column 2: Equation should read as follows:

$$C = \frac{V_o - V}{V_o}; \quad \frac{P}{C} = \frac{1}{ab} + \left(\frac{1}{a}\right) P$$

Page 5, Column 1: "Vo = initial volume" should read
"V_o = initial volume"

THE FRICTION INDEX AND PRESSING CHARACTERISTICS
OF SPHERICAL STAINLESS STEEL POWDER AND OXIDIZED COPPER POWDER

by

H. Sheinberg

ABSTRACT

A procedure is described for accumulating data on the friction index and compression ratio of a copper powder at different stages of oxidation and screened fractions of a stainless steel powder. The dimensionless and nameless powder property, friction index divided by the compression ratio, under certain conditions, was constant for a regular series of size fractions of stainless steel powder. Calculations were made of the coefficient of compression and the limiting value of the degree of volume change in the manner of Kawakita.

INTRODUCTION

Hausner has suggested¹ that the ratio of the tap density (ρ_T) to the bulk density (ρ_B) of a powder may index the powder's friction characteristics. He obtained a constant for a regular series of size fractions of stainless steel powder by dividing the friction index of each specific fraction by the compression ratio of that fraction.

At the time of Hausner's suggestion, I believed that a constant should be obtained for this ratio only in the relatively flat portion (high-pressure region) of the density versus pressure curve. To verify this assumption, I measured the ρ_B and ρ_T for a regular $\sqrt[4]{2}$ screen size series of pseudo-spherical stainless steel powder, determined the compression ratio of each fraction at varying pressure, and calculated values of this nameless powder property. To ascertain how oxidation influenced this nameless property, I determined the ρ_B , ρ_T , and compression ratio of freshly reduced copper powder and of this copper powder at various

stages of oxidation.

I also used the accumulated data for making calculations of the coefficient of compression and the limiting value of degree of volume change as outlined by Kawakita² for studying volume decrease under pressure.

STAINLESS STEEL

Pressing Procedure

A -100 mesh, atomized type 316 stainless steel powder shown in Fig. 1 and purchased from the Federal-Mogul Corporation was screened into the following fractions (U.S.S.): -100 +140; -140 +170; -170 +200; -200 +230; -230 +270; -270 +325; and -325 +400. Figure 2 shows weight percent distribution of the fractions. The bulk and tap densities were determined using a 10-ml graduated cylinder with a conical top.

A 15-g charge of each fraction was loaded into a double acting, 0.625-in. i.d., hardened steel die, which had been lubricated with cetyl alcohol. The die was positioned in a 20-ton Elmes hydraulic

Fig. 1. Atomized type 316 stainless steel powder, 250X.

up-acting press with a 3.0-in.-diam ram. Hydraulic pressure was monitored on a 0 to 500-psi Heise gauge with 1-psi graduations or on a 0 to 5000-psi Heise gauge with 10-psi graduations. Press platten movement was measured with a 2-1/2-in.-diam dial gauge graduated in 0.001 in. The dial gauge was zeroed at 25-psig; 20-psig was required to raise the press platten.

The pressure was incrementally increased by hand pumping according to the schedule in Table I and dial readings were recorded.

TABLE I

SCHEDULE OF GAUGE AND PUNCH PRESSURES

Gauge Pressure (psig)	Pressure on Die Punch (psi)	(tsi)
25	575	0.29
50	1,150	0.58
100	2,300	1.16
250	5,750	2.88
400	9,200	4.60
600	13,800	6.80
900	20,750	10.38
1200	27,600	13.80
1500	34,500	17.25
1800	41,400	20.70
2100	48,300	24.15
2400	55,200	27.60
2700	62,100	31.05

Because compacted, unlubricated, spherical stainless steel powder has very little strength, the length of the pressed powder compact was determined by measuring the total length of the punches

Fig. 2. Screen analysis of +400 mesh type 316 stainless steel powder.

plus compact before ejection.

Data

The measured and calculated powder properties are presented in Table II.

The friction index, ρ_T/ρ_B , divided by the compression ratio, l_i/l_f , gives a constant; however, it should be pointed out that 1) the numbers listed for ρ_B and ρ_T are probably only accurate to 0.1 g/cc and 2) for Table I, l_i is calculated based on ρ_B and therefore contributes to obtaining a constant.

The values for this ratio are not as constant in the rapidly ascending portion of the density versus pressure curve. A typical pressure versus density curve is shown in Fig. 3. Table III shows values of the friction index divided by the compression ratio when the final length, l_f , was measured at only 100 and 900 psig. For comparison Table III shows the values of this ratio using the measured l_i at 25 psig. Note the wide variations in the constant.

Kawakita derived the following equation relating the degree of volume change, C , to the pressure, P :

$$C = \frac{V_0 - V}{V_0} + \frac{1}{ab} = \left(\frac{1}{a}\right)P,$$

TABLE II
DATA ON PSEUDO-SPHERICAL STAINLESS STEEL POWDER

Fraction (U.S.S.)	Distribution (wt %)	ρ_B (g/cc)	ρ_T (g/cc)	ρ_T/ρ_B	l_i (in.)	l_f (in.)	l_i/l_f	$\rho_T/\rho_B / l_i/l_f$
-100 +140	40.1	4.53	5.02	1.108	0.659	0.469	1.41	0.79
-140 +170	12.4	4.54	5.07	1.115	0.657	0.469	1.40	0.79
-170 +200	9.2	4.53	5.12	1.130	0.659	0.462	1.43	0.79
-200 +230	1.2	4.40	5.04	1.145	0.678	0.469	1.44	0.79
-230 +270	16.5	4.48	5.00	1.117	0.666	0.475	1.40	0.80
-270 +325	9.4	4.43	5.08	1.147	0.674	0.468	1.44	0.79
-325 +400	11.2	4.46	5.07	1.137	0.669	0.466	1.43	0.81

ρ_B - bulk density.

l_i - initial length.

l_i/l_f - compression ratio.

ρ_T - tap density.

l_f - final length at 2700 psig.

ρ_T/ρ_B - friction index.

Fig. 3. Typical density versus pressure curve for type 316 stainless steel powder.

where

V_o = initial volume,
 V = final volume,
 a = limiting value of C at high pressure,
 b = the coefficient of compression related to the rheological nature of the powder.

Kawakita found this equation for a straight line to hold for a variety of powders from talc to TiO_2 .

The data obtained in pressing the fractions of stainless steel powder were used for making calculations in the manner of Kawakita. Figure 4 is a plot of these calculations, and the slope ($1/a$) and intercept ($1/ab$) can be determined assuming a con-

TABLE III
FRICTION INDEX/COMPRESSION RATIO AT THREE PRESSURES

Fraction (U.S.S.)	$\rho_T/\rho_B / l_i/l_{100}$	$\rho_T/\rho_B / l_i/l_{900}$	$\rho_T/\rho_B / l_{25}/l_{2700}$
-100 +140	1.05	0.93	0.80
-140 +170	1.06	0.94	0.83
-170 +200	1.11	0.91	0.86
-200 +230	1.05	0.93	0.85
-230 +270	1.10	0.95	0.82
-270 +325	1.05	0.96	0.86
-325 +400	1.15	1.01	0.78

tinuous straight line. The data used for making these plots are presented in Table IV. However, if the data obtained at low pressures is included in this plot, the result is that shown (for one fraction) in Fig. 5. The straight portion of the curve is extrapolated to determine the intercept.

The negative deviation from a straight line (Fig. 5) is anticipated because with a large variety of powders the initial high rate of densification at low pressure is primarily a rearrangement of particles, whereas the densification at high pressure involves primarily plastic deformation of the particles. Depending on the nature of the material, fracturing may also occur under high pressures.

Kawakita states that for a large variety of powders the value of initial pore fraction divided

Fig. 4. P/C versus P for fractions of type 316 stainless steel powder.

TABLE IV
DATA FOR KAWAKITA PLOTS

Mesh	l_{25} (in.)	V_{25} (cc)	l_f (in.)	V_f (cc)	$V_{25}-V_f$ (cc)	$(V_{25}-V_f)100/V_{25}$	P/C
<u>at 31.05 tsi</u>							
-100 +140	0.646	3.25	0.469	2.36	0.89	27.4	1.135
-140 +170	0.638	3.21	0.469	2.36	0.85	26.5	1.172
-170 +200	0.604	3.04	0.462	2.33	0.71	23.4	1.33
-200 +230	0.634	3.19	0.469	2.36	0.83	26.0	1.195
-230 +270	0.648	3.26	0.475	2.39	0.87	26.7	1.16
-270 +325	0.628	3.16	0.468	2.35	0.81	25.7	1.203
-325 +400	0.687	3.46	0.466	2.35	1.11	32.1	0.970
<u>at 17.25 tsi</u>							
-100 +140	0.646	3.25	0.515	2.59	0.66	20.3	0.850
-140 +170	0.638	3.25	0.517	2.60	0.61	19.0	0.908
-170 +200	0.604	3.04	0.502	2.52	0.52	17.1	1.01
-200 +230	0.634	3.19	0.515	2.59	0.60	18.8	0.917
-230 +270	0.648	3.25	0.524	2.63	0.63	19.3	0.894
-270 +325	0.628	3.16	0.515	2.59	0.57	18.0	0.968
-325 +400	0.627	3.46	0.533	2.68	0.78	22.5	0.766

Fig. 5. Kawakita plot of P/C versus P for -100 +140 mesh, type 316 stainless steel powder.

by 0.40 is near unity for spherical powders and becomes smaller than unity when departure from sphericity is increased. This value of 0.40 is derived by assuming one-size spheres and an eight-neighbor contact for each spherical particle and calculating, therefore, a theoretical void of 39.54%. Table V includes the data for the various size fractions calculated in the manner of Kawakita. The values of (pore fraction/0.40) are very close to unity, as Kawakita predicts. One would expect a departure from unity with spherical powders when there is a wide size distribution of the spheres.

Kawakita concludes that at high pressures the value of "a" should be approximately that of the initial porosity. Table V shows no close agreement

between these two values but they are within the same range.

COPPER

Pressing Procedure

Grade NF-IM copper powder (Sheritt Gordon Mines, Ltd.) was freshly reduced in H_2 at $360^\circ C$ and screened -325 mesh. The bulk and tap densities were determined using a 10-ml graduated cylinder with a conical top. The average particle size was determined with the Fisher subsieve sizer. A 8.95-g charge was loaded into a double acting, 0.500-in. i.d., hardened steel die and pressed in the same manner as the stainless steel powder. The pressure was incrementally increased by hand pumping according to the schedule in Table VI and dial readings were recorded. The compact was then ejected, weighed, and measured.

TABLE VI

SCHEDULE OF GAUGE AND PUNCH PRESSURES

Gauge Pressure (psig)	Pressure on Die (psi)	Punch (tsi)
25	900	0.45
50	1,500	0.75
100	3,600	1.8
250	9,000	4.5
450	16,200	8.1
600	21,600	10.8
900	32,400	16.2
1200	43,200	21.6
1500	54,000	27.0
1800	64,800	32.4
2100	75,600	37.8

The container with the balance of the freshly reduced copper was weighed analytically and the powder oxidized in steps, either at room temperature,

TABLE V

DATA CALCULATED IN THE MANNER OF KAWAKITA

Size Fraction	Intercept	a	b	Initial Porosity	Pore Fraction	Pore Fraction/0.04
-100 +140	0.507	48.3	0.0415	42.6	0.426	1.06
-140 +170	0.580	52.3	0.0329	42.5	0.425	1.04
-170 +200	0.612	43.3	0.0370	42.6	0.426	1.06
-200 +230	0.573	49.7	0.0351	44.3	0.443	1.11
-230 +270	0.565	52.1	0.0341	43.2	0.432	1.08
-270 +325	0.670	57.8	0.0258	43.8	0.438	1.09
-325 +400	0.517	68.1	0.0284	43.6	0.436	1.09

75°, or 85°C in air. After each oxidation step the weight gain was determined by analytical weighing; the ρ_B , ρ_T , and Fisher subsieve size were determined; and a compact was pressed with the oxidized powder.

Fig. 6. P/C versus P for copper powders at various stages of oxidation.

Data

Table VII summarizes the data for the copper powder. The ρ_T/ρ_B fits a predictable pattern through the first two stages of oxidation (through 0.137 wt % increase): the data indicates reduced friction with increased oxidation. It is possible that past this stage the type of oxide film changed or became thick and flaked off. In any event a constant is not obtained for ρ_T/ρ_B l_i/l_f whether the l_i calculated from ρ_B or the actual l_i measured at 25 psig is used.

The Kawakita-type plots shown in Fig. 6 are derived from the data of Table VII. The slope was calculated and the intercept determined by extrapolating the straight lines. Figure 7 shows the slight deviation from a straight line at low pressures that was typical for the copper pressings. It is probable that this deviation is not very marked because the soft copper powder deforms slightly, even at low pressure.

Table VIII includes values of a, b, and initial porosity. Although there is no agreement between the values of "a" and initial porosity, they are in the same range.

DISCUSSION

For copper powder at various stages of oxidation the data followed a predicted decrease in friction index through the first two oxidation stages. It is possible that during the later stages of oxidation the type of oxide film on the particles changed or became very thick and flaked. The value of the friction index divided by the compression ratio was not constant but increased with increasing oxidation.

TABLE VII

DATA ON PRESSING REDUCED AND OXIDIZED COPPER POWDER

Cumulative Increase (wt %)	ρ_B (g/cc)	ρ_T (g/cc)	ρ_T/ρ_B	FSS (μ)	ρ_F (g/cc)	l_i (in.)	l_f (in.)	l_i/l_f (calc.)	$\rho_T/\rho_B / l_i/l_f$	l_{25} (in.)	l_{25}/l_f	$\rho_T/\rho_B / l_{25}/l_f$
As reduced	1.21	1.86	1.537	1.81	7.26	2.300	0.382	6.02	0.255	1.341	3.52	0.436
0.100	1.28	1.90	1.485	1.83	7.28	2.170	0.380	5.71	0.260	1.339	3.51	0.425
0.137	1.40	1.99	1.420	1.86	7.08	1.985	0.391	5.08	0.279	1.418	3.63	0.392
0.199	1.42	2.08	1.465	1.87	7.07	1.955	0.391	5.00	0.292	1.437	3.68	0.398
0.289	1.49	2.18	1.464	1.89	7.03	1.865	0.394	4.47	0.308	1.505	3.82	0.380

ρ_B - bulk density.
 ρ_T - tap density.
 ρ_F - pressed density.

FSS - Fisher subsieve sizer average particle size (μ) at 0.778 P.
 l_i - initial calculated length based on ρ_B .
 l_{25} - initial length at 25 psig.

Fig. 7. P/C versus P for copper oxidized to 0.289 wt % increase.

TABLE VIII

INITIAL POROSITY AND KAWAKITA VALUES FOR COPPER

Cumulative Increase (wt %)	a	b	Initial Porosity
As reduced	79.5	0.292	87.5
0.100	76.3	0.306	85.7
0.137	81.9	0.244	84.4
0.199	80.0	0.260	84.2
0.289	80.6	0.264	83.4

For the regular series size fractions of stainless steel powder, a constant is obtained for the ratio of the friction index divided by the compression ratio at high pressures. Inasmuch as the value of this ratio is a function of the pressure at which the data is accumulated, a single value for this constant should not be considered as a characteristic of the pressing behavior of a specific powder or size fraction thereof.

Although the data, when plotted in the manner described by Kawakita, can be used to calculate the value of "a" and "b" in the formula $C = 1/ab + (1/a)P$, I doubt that a straight line plot is obtained for this relationship for all pressure ranges. It is generally agreed that densification during pressing of powders occurs in at least two stages and by at least two wholly different mechanisms. I believe that this would preclude a single straight line describing change in volume during pressing. The data in Fig. 5 more accurately describes the relationship between volume change and

pressure. This would suggest that the data in Kawakita's straight-line plots are extrapolations at low pressure.

The calculated values of "a" did not closely agree with the measured values of initial porosity as predicted by Kawakita, but they were in the same range.

Kawakita's prediction that the pore fraction divided by 0.40 should be approximately unity for spherical particles was verified for all size ranges of the type 316 pseudo-spherical particles. This value varied from 1.04 to 1.11.

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