

Letter

A study of a new phenomenological compacting equation

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Abstract

A new phenomenological compacting equation has been proposed. The equation is $\ln(1/(1-D)) = A\sqrt{P} + B$ where P is the applied pressure, D is the relative density of the compact, A is a parameter related to densification of the compact by plastic deformation, and B is a parameter related to powder density at the start of compaction. Linear regression analysis has been used to compare the new equation with the four compacting equations often used and proposed by Balshin, Heckel, Kawakita and Ge. The results show that the new equation gives linear correlation coefficients very close to unity. This equation, together with parameters A and B , permits improved evaluation of the compacting characteristics, compared to that performed by other equations. © 2001 Elsevier Science S.A. All rights reserved.

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1. Introduction

The powder compaction process plays an important role in the manufacture of a variety of products that include ceramics, metallic parts, fertilizers and pharmaceuticals [1]. In the case of ceramics and metallic parts, the microstructure of sintered powder compacts depend strongly on the quality of the green compact, which in turn depends on the behavior of the powder during compaction, that is, by the density–pressure relationship of the powder [2].

According to Bockstiegel [3], the interest originally in density–pressure relationships stemmed from the practical problem of being able to predict the pressures required for achieving a certain density, later on, the interest seemed to shift more towards the analytical problem of finding an adequate but simple mathematical description for experimentally obtained density–pressure curves. Considerable effort has been made to characterize powders and their compaction behavior using a compacting equation [2,4–10].

In this paper the four often used compaction equations are compared, in a manner similar to that carried out by Ge

[8] in 1995. A new phenomenological compacting equation has also been included in this paper [9].

2. Compacting equations

Balshin attempted to correlate the relative density of compact powders with externally applied pressures and proposed the following equation:

$$\frac{\partial D}{\partial P} = k \frac{D^2}{P} \quad (1)$$

$$\ln P = -\frac{A_1}{D} + B_1 \quad (2)$$

where P is the applied pressure, D is the relative density of the compact and A_1 , B_1 are constants. Balshin called A_1 the pressing modulus and considered it to be analogous to the Young's modulus [2].

Balshin's equation can be rewritten as:

$$\frac{1}{D} = A_2 \ln P + B_2 \quad (3)$$

Heckel, in 1961 [5], considered the compaction of powders to be analogous to a first-order chemical reaction. The pores are the reactant and the densification of the bulk

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is the product. The “kinetics” of the process may be described as a proportionality between the change in density with pressure and the pore fraction by:

$$\frac{\partial D}{\partial P} = K(1 - D) \quad (4)$$

where $(1 - D)$ is the pore fraction and K is a constant. The final form of Heckel’s equation can be written as follows:

$$\ln\left(\frac{1}{1 - D}\right) = A_3 P + B_3 \quad (5)$$

where A_3 and B_3 are constants. Heckel postulated that the constant A_3 gave a measure of the ability of the compact to densify by plastic deformation and constant B_3 represented the degree of packing achieved at low pressures as a result of rearrangement before appreciable amounts of inter-particle bonding occurred.

Kawakita and Lüdde [6] derived a compaction equation from the observed relationship between applied pressure and volume or relative density. Kawakita obtained the following equation:

$$\frac{D}{D - D_0} = \frac{A_4}{P} + B_4 \quad (6)$$

where A_4 and B_4 are constants and D_0 the relative powder apparent density without application of pressure.

Attention must be paid to the experimental determination of D_0 , and deviations from Kawakita’s equations are sometimes due to fluctuations in the measured value of D_0 , which is usually inaccurate.

Ge [1] proposed a new differential equation for the pressing of powders:

$$\frac{\partial D}{\partial P} = K \frac{(1 - D) D^n}{P^m} \quad (7)$$

where n and m are constants.

From Eq. (7), Ge obtained the following equation:

$$\log\left[\ln\left(\frac{1 - D_0}{1 - D}\right)\right] = A_5 \log P + B_5 \quad (8)$$

Ge, in a second paper published in 1995 [8], proposed a further simplified form of Eq. (8). He observed that his equation was subject to the same level of imprecision as Kawakita’s equation, due to fluctuations in the measured value of D_0 . Ge considered that D_0 approached zero when total pressure reached zero, and his equation could be rewritten as:

$$\log\left[\ln\left(\frac{1}{1 - D}\right)\right] = A_6 \log P + B_6 \quad (9)$$

where A_6 and B_6 are constants.

This simplification done by Ge in his equation is not theoretically solved.

In 1998, Panelli and Ambrozio Filho [9], based upon observations of typical compaction curves, proposed the following differential equation to represent such curves:

$$\frac{\partial q}{\partial P} = \frac{Kq}{P^m} \quad (10)$$

where q is relative porosity, P the applied pressure and K , m are constants.

By integrating Eq. (10) from q (porosity at P) to q_0 (porosity at $P = 0$) at applied pressures from 0 to P , a general equation is obtained:

$$\ln \frac{q_0}{q} = A_7 \frac{P^{-m+1}}{-m+1} + B_7. \quad (11)$$

This equation has an integration constant B_7 , which must be zero to satisfy the condition $q_0 = q$ when $P = 0$.

Fitting experimental data to it by regression analysis tested Eq. (11). Linear correlation coefficients, R , give values better than 0.99 when $m = 0.5$. So the value 0.5 was adopted for m in Eq. (10) and rewritten as:

$$\ln \frac{q_0}{q} = A_g \sqrt{P} \quad (12)$$

where $q_0 = (1 - D_0)$, $q = (1 - D)$ and D_0 and D are the relative densities of the powder at zero pressure and of the compact at pressure P .

Upon substitution, Eq. (12) becomes:

$$\ln\left[\frac{(1 - D_0)}{(1 - D)}\right] = A_g \sqrt{P}. \quad (13)$$

Eq. (13) has limited applicability owing to the necessity to determine the parameter D_0 . It is therefore important to

Table 1

The compacting equations that have been compared. P = applied pressure, D = relative density of the compact, D_0 = relative apparent density of powder in absence of applied pressure, and A_n and B_n are constant parameters; $n = 2, 3, 4, 6$ and 10

Author	Equation	Equation number	Reference
Balshin	$1/D = A_2 \ln P + B_2$	(3)	[4]
Heckel	$\ln(1/(1 - D))$ $= A_3 P + B_3$	(5)	[5]
Kawakita	$D/(D - D_0)$ $= (A_4/P) + B_4$	(6)	[6]
Ge	$\log[\ln(1/(1 - D))]$ $= A_6 \log P + B_6$	(9)	[8]
Panelli and Ambrozio Filho	$\ln(1/(1 - D))$ $= A_{10} \sqrt{P} + B_{10}$	(15)	[9]

Table 2
 Experimental data on the compaction of various powders. *P* = applied pressure (MPa) and *D* = relative density of compact

Powder no.	Material	Reference	Experimental data on compaction of powders													
1	Silver bromide, 8–10 mesh, 95°C	[4]	<i>P</i>	21	31	47	61	78	94	110						
			<i>D</i>	0.800	0.841	0.908	0.949	0.971	0.985	0.988						
2	Ammonium nitrate	[11]	<i>P</i>	1.0	1.9	3.0	4.7	7.2	11.6	19.6						
			<i>D</i>	58.8	0.625	0.667	0.714	0.769	0.833	0.909						
3	Silver bromide, 10–14 mesh, 25°C	[4]	<i>P</i>	21	31	47	61	78	94	110	126	142				
			<i>D</i>	0.740	0.789	0.861	0.909	0.934	0.954	0.967	0.977	0.985				
4	Silver bromide, 8–10 mesh, 25°C	[4]	<i>P</i>	21	31	47	61	78	94	110	126	142				
			<i>D</i>	0.743	0.791	0.858	0.905	0.934	0.954	0.969	0.977	0.980				
5	Silver bromide, 14–20 mesh, 25°C	[4]	<i>P</i>	21	31	47	61	78	94	110	126	142				
			<i>D</i>	0.743	0.781	0.852	0.900	0.927	0.954	0.965	0.976	0.980				
6	Atomized tin	[1]	<i>P</i>	0	14.7	19.6	29.4	49.0	68.6	98.0	117.6	147.0	196.0	245.0	294.0	
			<i>D</i>	0.501	0.739	0.799	0.853	0.920	0.951	0.975	0.984	0.990	0.993	0.996	0.997	
7	Lead (metallic)	[11]	<i>P</i>	2.1	5.1	8.9	13.4	20.5	32.5							
			<i>D</i>	0.625	0.667	0.714	0.769	0.833	0.909							
8	Atomized lead	[1]	<i>P</i>	0	32.34	64.68	96.04	130.3	160.7	196.0						
			<i>D</i>	0.439	0.937	0.974	0.986	0.991	0.993	0.995						
9	Ammonium Chloride	[11]	<i>P</i>	0.7	0.9	1.6	2.2	2.9	4.0	5.5	8.0	12.1	18.7	32.0	63.7	
			<i>D</i>	0.455	0.476	0.500	0.526	0.556	0.588	0.625	0.667	0.714	0.769	0.833	0.909	
10	Potassium chloride	[11]	<i>P</i>	1.2	1.9	2.6	3.6	4.9	6.6	8.9	11.7	15.9	21.3	29.1	42.1	76.9
			<i>D</i>	0.435	0.455	0.476	0.500	0.526	0.556	0.588	0.625	0.667	0.714	0.769	0.833	0.909
11	Potassium nitrate	[11]	<i>P</i>	0.6	2.1	4.4	7.4	11.6	17.8	26.8	41.2	76.4				
			<i>D</i>	0.526	0.556	0.588	0.625	0.667	0.714	0.769	0.833	0.909				
12	Sodium chloride	[11]	<i>P</i>	4.3	7.0	10.2	14.8	21.4	31.2	45.0	65.9	101.9				
			<i>D</i>	0.526	0.556	0.588	0.625	0.667	0.714	0.769	0.833	0.909				
13	Barium nitrate	[11]	<i>P</i>	0.7	1.5	2.7	4.7	9.2	22.3	68.6						
			<i>D</i>	0.556	0.588	0.625	0.667	0.714	0.769	0.833						
14	Electrolytic copper	[1]	<i>P</i>	0	59.0	108	114	187	294	372	1000					
			<i>D</i>	0.300	0.607	0.664	0.674	0.753	0.839	0.874	0.964					
15	Stainless steel	[1]	<i>P</i>	0	154.8	308.7	463.5	617.4	772.2	926.1						
			<i>D</i>	0.374	0.648	0.740	0.800	0.831	0.870	0.890						
16	AISI M2	[9]	<i>P</i>	260.4	365.6	474.3	567.3	655.7								
			<i>D</i>	0.620	0.677	0.716	0.755	0.778								
17	Calcium carbonate	[11]	<i>P</i>	1.9	3.1	4.8	7.4	12.3	21.9	41.2	86.0	178.2				
			<i>D</i>	0.435	0.455	0.476	0.500	0.526	0.556	0.588	0.625	0.667				
18	Spray-dried alumina	[12]	<i>P</i>	14	28	49	69	96	140	206	278	343	441	447	481	503
			<i>D</i>	0.511	0.557	0.580	0.609	0.625	0.650	0.673	0.694	0.695	0.713	0.714	0.716	0.722
19	M2 + 10 vol% NbC, mechanical alloyed 5h	[10]	<i>P</i>	260	365	474	567	655								
			<i>D</i>	0.603	0.631	0.650	0.673	0.690								
20	Tungsten carbide	[1]	<i>P</i>	0	49.0	68.6	98.0	147	196	245	294	392	490	588		
			<i>D</i>	0.201	0.471	0.489	0.512	0.538	0.559	0.576	0.586	0.609	0.627	0.641		
21	Titanium carbide	[1]	<i>P</i>	0	18.0	36.0	72.0	144	288	576	864					
			<i>D</i>	0.370	0.518	0.547	0.587	0.614	0.650	0.699	0.719					
22	Alumina	[9]	<i>P</i>	365.6	474.3	567.3	655.7									
			<i>D</i>	0.502	0.513	0.521	0.528									

Table 3
 Linear regression analysis of the five-compaction equations. R = linear regression coefficient. A_n and B_n are the equations coefficients, where $n = 2, 3, 4, 6$ and 10

Powder no.	Eq. (3)			Eq. (5)			Eq. (6)			Eq. (9)			Eq. (15)		
	A_2	B_2	R	$A_3 (\times 10^{-2})$	B_3	R	A_4	B_4	R	A_6	B_6	R	A_{10}	B_{10}	R
1	-0.1520	1.7017	-0.983	0.0338	0.8635	0.995				0.6520	-0.6855	0.992	0.5102	-0.9275	0.992
2	-0.2080	1.7179	-0.998	0.0802	0.8484	0.998				0.3317	-0.0947	0.978	0.4381	0.3591	0.991
3	-0.1780	1.8672	-0.990	0.0233	0.8810	0.999				0.6015	-0.6923	0.996	0.3864	-0.5871	0.994
4	-0.1751	1.8556	-0.982	0.0222	0.9383	0.996				0.5858	-0.6670	0.996	0.3698	-0.4758	0.996
5	-0.1787	1.8752	-0.985	0.0221	0.9017	0.997				0.5866	-0.6760	0.994	0.3674	-0.4985	0.994
6	-0.1077	1.5589	-0.924	1.6113	1.6838	0.963	16.398	1.9063	0.991	0.4998	-0.4424	0.997	0.3474	0.1267	0.995
7	-0.1868	1.7751	-0.988	0.0467	0.8549	0.999				0.3186	-0.1601	0.952	0.3321	0.3584	0.975
8	-0.0339	1.1765	-0.950	1.4920	2.5854	0.971	3.6059	1.7681	0.996	0.3612	-0.0962	0.997	0.3027	1.1676	0.993
9	-0.2546	2.0759	-0.993	0.0283	0.7560	0.971				0.3018	-0.2114	0.989	0.2500	0.3886	0.999
10	-0.3098	2.3783	-0.997	0.0246	0.6438	0.988				0.3448	-0.3393	0.979	0.2373	0.2256	0.994
11	-0.1766	1.9003	-0.983	0.0218	0.8148	0.993				0.2386	-0.1615	0.940	0.2086	0.4586	0.988
12	-0.2637	2.3100	-0.999	0.0164	0.7265	0.999				0.3921	-0.4570	0.983	0.1984	0.2361	0.986
13	-0.1350	1.7343	-0.992	0.0130	0.9799	0.916				0.1778	-0.0758	0.998	0.1305	0.7753	0.982
14	-0.2302	2.5584	-0.977	0.2521	0.9122	0.985	34.031	1.4547	0.974	0.4700	-0.8999	0.993	0.1032	0.0516	0.998
15	-0.2350	2.7125	-0.995	0.1493	0.8641	0.995	117.86	1.6157	0.997	0.4179	-0.9041	0.997	0.0647	0.2189	0.998
16	-0.3550	3.5815	-0.998	0.1360	0.6208	0.998				0.4779	-1.1771	0.998	0.0568	0.0431	0.998
17	-0.1769	2.3772	-0.994	0.0027	0.6730	0.906				0.1447	-0.2858	0.999	0.0435	0.5638	0.977
18	-0.1510	2.3096	-0.992	0.0959	0.8404	0.942				0.1599	-0.3251	0.998	0.0276	0.6816	0.984
19	-0.2216	2.8919	-0.996	0.0616	0.7667	0.998				0.2509	-0.6426	0.992	0.0256	0.0506	0.995
20	-0.2278	3.0013	-0.999	0.0699	0.6512	0.974	12.259	1.4645	0.970	0.1924	-0.5260	0.999	0.0224	0.4934	0.997
21	-0.1393	2.3225	-0.998	0.0598	0.8818	0.950	25.406	2.2157	0.959	0.1434	-0.3225	0.997	0.0212	0.6768	0.991
22	-0.1673	2.9797	-0.999	0.0184	0.6308	0.998				0.1262	-0.4805	0.999	0.0083	0.5378	1.000

simplify Eq. (13) by eliminating D_0 . This can be done as follows:

$$\ln\left(\frac{1}{1-D}\right) = A_9\sqrt{P} + \ln\left(\frac{1}{1-D_0}\right) \quad (14)$$

$$\ln\left(\frac{1}{1-D}\right) = A_{10}\sqrt{P} + B_{10} \quad (15)$$

where A_{10} and B_{10} are constants characteristics of the powder [9]. Constants A_{10} and B_{10} can be used as parameters to characterize the powder behavior during pressing. It can be stated that parameter A_{10} (inclination of the compressibility curve) provides the plastic deformation capacity of the powder in compaction. Thus, as A_{10} increases, the powder undergoes increasing plastic deformation during compaction. It is also postulated that parameter B_{10} (interception of the curve resulting from Eq. (15) at zero pressure) expresses the density in the absence of pressure.

Table 1 presents the compacting equations that have been compared.

3. Linear regression analysis

The compaction equations proposed by Balshin, Eq. (3), by Heckel, Eq. (5), by Kawakita, Eq. (6), by Ge, Eq. (9), and by Panelli and Ambrozio Filho, Eq. (15), can be rewritten in a general form as:

$$f_1(D) = A_n f_2(P) + B_n \quad (16)$$

where $f_1(D)$, $f_2(P)$, A_n and B_n are dependent on the equations, so that $f_1(D) = 1/D$, $f_2(P) = \ln P$, A_2 and B_2 for Balshin's equation, $f_1(D) = \ln[1/(1-D)]$, $f_2(P) = P$, A_3 and B_3 for Heckel's equation, $f_1(D) = D/(D-D_0)$, $f_2(P) = 1/P$, A_4 and B_4 for Kawakita's equation, $f_1(D) = \log \ln[1/(1-D)]$, $f_2(P) = \log P$, A_6 and B_6 for Ge's equation, $f_1(D) = \ln[1/(1-D)]$, $f_2(P) = \sqrt{P}$, A_{10} and B_{10} for Panelli and Ambrozio Filho's equation.

If the values of $f_1(D)$ are plotted against the values of $f_2(P)$, a straight-line relationship will be attained for each of the compaction equations. Thus, a quantitative comparison between the equations can be made by linear regression analysis. Different sets of experimental data on compaction are shown in Table 2. Eqs. (3), (5), (6), (9) and (15) tested each set of data and the results are shown in Table 3. Both tables are constructed ordering A_{10} values of Eq. (15) in a decreasing way from top to bottom of the tables.

4. Discussion

Due to the non-availability of D_0 only a fraction of the data sets presented in Table 2 could be tested with Eq. (6) as shown in Table 3. The application of Eq. (6) is quite limited, because the equation can be used only when D_0 ,

the relative density of the loose powder at zero pressure, is known. The other four equations need only the parameters D , relative density of the compact and P , the applied pressure.

The correlation coefficients, R , (as shown in Table 3) for the five equations analyzed have a mean value and range of variation as follow: Eq. (3) mean: 0.986, range: 0.924 to 0.999; Eq. (5) mean: 0.979, range: 0.906 to 0.999; Eq. (6) mean: 0.981, range: 0.970 to 0.997; Eq. (9) mean: 0.989, range: 0.940 to 0.999; and Eq. (15) mean: 0.993, range: 0.975 to 0.999. From these values it can be concluded that Eq. (15) shows the highest mean correlation coefficient, R , and narrowest range of variation. Furthermore, the lowest value of R found by Eq. (15) is 0.975, which is higher than the lowest values of R when using (Eqs. (3), (5), (6) and (9)). Nevertheless, all the equations provide reasonable fits and could be used.

The equations can be evaluated taking into consideration that two processes are usually involved in the compaction: particle rearrangement followed by plastic deformation and/or fragmentation. Plastic deformation is the main mechanism in pressing of ductile powders and fragmentation is the main mechanism in pressing of brittle powders. Generally, rearrangement occurs at pressures less than 1 MPa and densification due to rearrangement is dependent on powder characteristics, but corresponding to the first 5% to 10% decrease in porosity. At higher pressures, plastic deformation or fragmentation is the major form of densification for powders.

A plot of $f_1(D)$ versus $f_2(P)$ from the general Eq. (16) for the five compacting equations would give two straight lines, one for smaller pressures corresponding to particle rearrangement and the other for higher pressures corresponding to plastic deformation or fragmentation of the particles. Fig. 1a shows a qualitative compression curve by plotting the relative density D of the powder against compacting pressure P and Fig. 1b shows the two straight lines by plotting $f_1(D)$ against $f_2(P)$.

As more than one mechanism can operate in the compaction, no single equation can perfectly represent the phenomenon over the complete range of pressures. On the other hand the rearrangement represents a small amount of the total densification, causing only a small error in the fit of the equation.

The significance of the two constants that appear in all the equations can also be considered.

As shown in the introduction, parameter A_{10} from Eq. (15) represents the powder's ability to densify by plastic deformation. Plastic materials, especially soft metals, with the highest A values are at the top of Table 2, and the brittle materials (ceramics) with the lowest A_{10} values are at the bottom of the table. This aspect can also be seen in Fig. 2, where some plots of \sqrt{P} versus $\ln(1/(1-D))$ using Eq. (15) are presented.

Eq. (5) has a format similar to that of Eq. (15), showing that the parameter A_3 of that equation would also show the

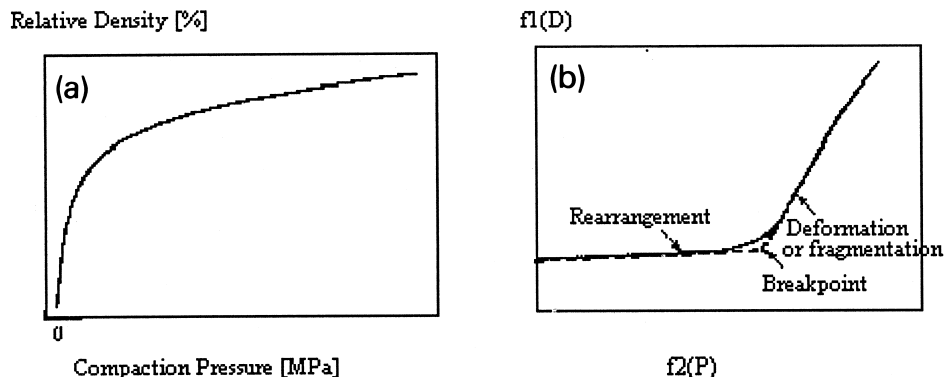


Fig. 1. (a) Qualitative compression curve of the relative density D of the powder against compacting pressure P and (b) schematic representation of $f_1(D)$ against $f_2(P)$, when plastic deformation or fragmentation follows particle rearrangement.

powder's ability to densify by plastic deformation. However, there is no similar order in the A_3 parameter, as shown by A_{10} in Eq. (15). The other equations also do not present a parameter that can be used to characterize the powder in the same way as A_{10} from Eq. (15).

Parameter B has no significance in (Eqs. (3), (6) and (9), because the equations are not defined at zero pressure.

In Eqs. (5) and (15), parameters B_3 and B_{10} can be used to calculate the relative density of the powder at zero pressure D_0 . Nevertheless, as pointed out earlier, at the beginning of compaction rearrangement must be considered (Fig. 1b). Therefore, D_0 calculated by using B_3 and B_{10} , would be different from the relative density of loose powder (D_{ap}), determined by ASTM B212 or similar.

In Heckel's equation [5], as he obtained B_3 always somewhat higher than $\ln(1/(1 - D_{ap}))$, it was considered

that D_0 would represent the degree of packing achieved at low pressures due to the rearrangement processes.

In the case of B_{10} of Eq. (15), their values are higher or lower than $\ln(1/(1 - D_{ap}))$ giving D_0 values higher or lower than D_{ap} , or even negative D_0 . The obtained values depend on the rearrangement and on inaccuracies in the regression analysis due to the use of high pressures in the determination of the curves (as discussed in a previous publication [9]). Considering that rearrangement represents only a small fraction of total densification in compaction [2], D_0 values calculated from Eq. (15) could be used as an approximation of the loose powder density (D_{ap}). Actually, for the real determination of D_{ap} it would be necessary to obtain the rearrangement equation.

The possibility to characterize the powders during compaction with the constants of Eq. (15) together with its

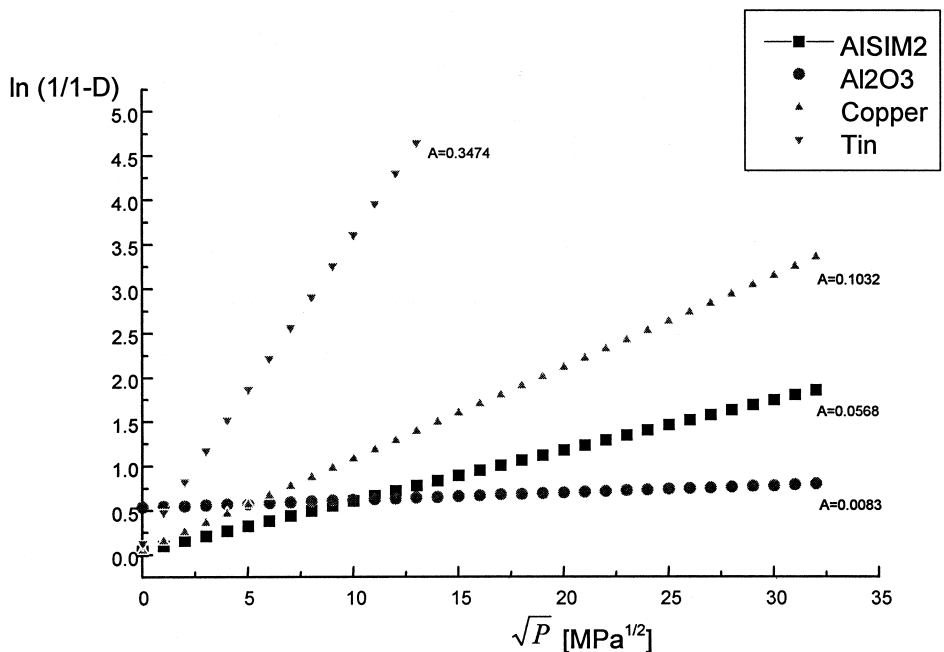


Fig. 2. Some compressibility curves.

good fit with experimental results are the main reasons to use this equation in modeling the compaction process.

5. Conclusions

A new phenomenological compaction equation is compared with the four often used compaction equations. From this comparison it is possible to conclude that: Eq. (15), i.e., $\ln(1/(1-D)) = A_{10}\sqrt{P} + B_{10}$, is the one which best represents the density–pressure relationship for powders. Its linear correlation coefficient is almost close to the unity.

Only Eq. (15) has constants representing powder characteristics during compaction: parameter A_{10} represents the ability of the powder to densify by plastic deformation and parameter B_{10} represents the density of the powder in the beginning of the compaction. However, due to the rearrangement mechanism at the beginning of compaction, B determined by the equation can be inaccurate.

6. List of symbols

A_n	Constants for n varying from 1 to 10
B_n	Constants for n varying from 1 to 10
D	Relative density of compact at pressure P
D_0	Relative powder density in absence of applied pressure

D_{ap}	Relative apparent density
K, k	Constants
m	Constant
P	Applied pressure [MPa]
q	Relative porosity of compact at pressure P
q_0	Relative porosity of compact in absence of applied pressure
R	Coefficient of linear regression

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