CONTRIBUTION TO ANALYSIS OF COMPACTION OF METAL POWDERS

Kaytbay S. H. M., Dudrová E.
Institute of Materials Research of SAS, Watsonova 47, 043 53 Košice, Slovakia

PRÍSPEVOK K ANALÝZE LISOVANIA PRÁŠKOVÝCH KOVOV

Kaytbay S. H. M., Dudrová E.
Ústav materiálového výskumu SAV, Watsonova 47, 043 53 Košice, Slovensko

Abstrakt

V práci je analyzované lisovanie práškového železa (ASC 100.29), legovaného Fe-Mo prášku (Astaloy 85 Mo) a zmesí na báze týchto práškov s prídavkom 3 hm.% medi a 0.7 hm.% grafitu. Pre kvantifikovanie závislosti pórovitosti od lisovacieho tlaku sa použili štyri lisovacie rovnice upravené do lineárneho tvaru $f_1(P)=a_n \cdot f_2(p)+b_n$ čo umožnilo porovnanie presnosti hodnotenia lisovacieho procesu podľa jednotlivých rovníc. Analýza vzťahu experimentálne nameraných a vypočítaných hodnôt pórovitosti ukázala, že najlepšie výsledky sa dosiahli v prípade lisovacej rovnice $P = P_o \cdot \exp\left(-K \cdot p^n\right)$ podľa Pariláka a kol. [10,11]. Táto rovnica umožňuje vypočítať parametre lisovateľnosti prášku, ako je celková práca "X_n", ktorá je potrebná na dosiahnutie „plnej hustoty" výlisku (P->0 at p->∞) premiestňovaním častíc prášku a ich plastickou deformáciou; práca "X_1", ktorá predstavuje prácu na zhustenie iba premiestnením častíc bez rozvoja plastickej deformácie; minimálny lisovací tlak "p_1", ktorý je potrebný pre dosiahnutie dostaočnej pevnosti surového výlisku. Výsledky ukázali, že hodnota tlaku p_1, je určená hlavne geometrickými vlastnosťami častíc, zatiaľ čo hodnoty X_n and X_1 závisia od plastických vlastností práškového kovu.

Abstract

The cold compaction in a tool of ferrous (ASC 100.29) and alloyed Fe-Mo (Astaloy 85 Mo) powders and the mixtures based on these powders with an addition of 3 wt. % of copper and 0.7 wt. % of graphite was quantified using four different compaction equations. A linear form of the dependence of porosity on pressing pressure $f_1(P)=a_n \cdot f_2(p)+b_n$ enables to compare the exactness of used compaction equations. The analysis of correlations between the experimentally determined porosity and calculated porosity showed that the best results were achieved in the case of the compaction equation $P = P_o \cdot \exp\left(-K \cdot p^n\right)$ given by Parilak et al [10, 11]. This equation enables to determine: the total work "X_n", which is necessary for achieving "full-dense" compact (P->0 at p->∞) by rearrangement of particles and by their plastic deformation; the work "X_1", which represents the part of the work for densification by particles rearrangement without plastic deformation; the minimal compaction pressure "p_1", necessary for achieving a sufficiently strength green compact. The results showed that the pressure p_1 is controlled mainly by morphology of powder particles, while the values of X_n and X_1 depend on plastic properties of metal powder.

Key words: powder metallurgy, powder compaction, quantification of compaction processes

1. Introduction

The powder compaction is an important part of the manufacture of sintered structural parts. From economical and technical point of view, the relation between the porosity (or density) of compact and pressing pressure gives information about the highest density (or lowest porosity) achieved at the lowest possible pressure. The metal powder compaction in a tool is described as multi-stage process [1]. At the beginning of a compaction cycle, the powder has a density approximately equal to the apparent density. As a pressure is applied, the first response is rearrangement of the particles with filling of large pores, giving a higher packing co-ordination. Increasing pressure provides better packing and leads to decreasing porosity with the formation of new particle contacts. High pressures increase density by contact enlargement through plastic deformation. Thus, the pressure causes localised deformation at the contacts, giving strain hardening and new particle contacts are formed. With an increasing of the applied pressure and with continued strain hardening of powder particles, the densification rate decreases, reflecting particles hardness. In a discussion of compaction the analyses of the green density or porosity dependence on the compaction pressure, so called „compaction equation“, is necessary.

A number of mathematical descriptions of the powder densification processes during the pressing in a tool were presented. Walker [2] and Balshin [3] correlated the relative density of pressed powders with an applied pressure p and presented the semilogratmic equation:

$$\ln(p) = -\frac{a_1}{D} + b_1 \tag{1}$$

D is relative density; a_1 and b_1 are constants. For $D = 1 - P$, where P is relative porosity, the equation (1) can be rewritten

$$\ln(p) = \frac{a_1}{(1-P)} + b_1 \tag{2}$$

According to Balshin, the constant a_1 is the pressing modulus and is analogous to the Young's modulus. Heckel [4] presented the density – compaction relationship with constants a_2 and b_2:

$$\ln\left[\frac{1}{(1-D)}\right] = a_2.p + b_2 \tag{3}$$

or
$$\ln\left(\frac{1}{p}\right) = a_2.p + b_2 \tag{4}$$

Kawakita and Lüdde [5] proposed the compaction equation from experimental values of the density achieved at the applied pressure p:

$$\frac{D}{(D-D_o)} = a_3 \cdot \left(\frac{1}{p}\right) + b_3 \tag{5}$$

or
$$\frac{(1-P)}{(P_o-P)} = a_3 \cdot \left(\frac{1}{p}\right) + b_3 \tag{6}$$

Where Po is the relative apparent density of the powder (for p=0).

Pamelli and Filho [6] expressed the compaction of powders by the equation:

$$\ln\left[\frac{(1-D_o)}{(1-D)}\right] = a_4 \cdot \sqrt{p} \tag{7}$$

or
$$\ln\left(\frac{P_o}{P}\right) = a_4 \cdot \sqrt{p} \tag{8}$$

or $$\ln(P)=\ln(P_o)-a_4\sqrt{p} \tag{9}$$

Ge [7] proposed a differential compaction equation, which can be expressed in the form:

$$\log\left[\ln\left(\frac{P_o}{(P-1)}\right)\right]=a_5\cdot\log(p)+b_5 \tag{10}$$

Shapiro [8] proposed a compaction formula of metal powders:

$$\ln(P)=\ln(P_o)-(a_6\cdot p)-\left(b_6\cdot\sqrt{p}\right) \tag{11}$$

Where the value of Po is porosity at zero external pressure and it is not identical with the apparent porosity [8, 9].

According to the Refs., [2-9], the constants "a" in the equations (1)-(11) represent a measure of the ability of powders to densification by plastic deformation, while the constants "b" are connected with the degree of particles arrangement at low pressures. In the References [2-7] did not analysed the relationship between the constants "a" and "b".

Parilák et al. [10, 11] proposed a compaction equation based on detailed analyses of a compaction curves for a set of more than 40 different metal powders. The authors [10,11] studied the interaction of geometrical and plastic properties of powder particles and their changes during during the compaction process. Their analyses resulted in description of compaction of metal powders by the formula:

$$P=P_o\cdot\exp\left(-K\cdot p^n\right) \tag{12}$$

or $$\ln\left[\ln\left(\frac{P_o}{P}\right)\right]=-\ln(K)+n\cdot\ln(p) \tag{13}$$

The equation (13) can be rewritten in a form:

$$\ln\left[\ln\left(\frac{P_o}{P}\right)\right]=a_7\cdot\ln(p)-\ln(b_7) \tag{14}$$

K and n in the equation (12) are the parameters with defined physico-metallurgical substance. Parameter K reflects the geometrical properties of particles and parameter n expresses the ability of particles to plastic deformation. The value of n for different powders is ranging from 0.5 to 1. In the case of powders with high plasticity, n is near to 0.5, in the case of a low plasticity, n is near to 1. The validity of the equation (12) was verified for the set of more than 40 powders [10, 11] and later, in the work [13] for a set of 81 powders [13] with different geometrical and metallurgical properties. The values of the parameters K and n were determined by regression analyses, while the correlation coefficients r of experimental compaction curves were ranged from 0.9884 to 0.9995. Based on the K and n values for different metal powders, the empirical relationship between the parameter K and n for the set of 81 powders was calculated [13]:

$$\ln(K)=1.401-7.7478\cdot n \tag{15}$$

with the correlation coefficient r = 0.9850. The equation (15) expresses the superposed effect of geometrical and metallurgical properties of particles during the compaction process – it means the continual changes of the geometry and strain hardening of particles with increasing pressing pressure. The solving of the equation (12) and (15) according to [10, 11] enables to calculate the total work "X_n", which is necessary for achieving of a full compact density (P->0 at p->∞) by rearrangement and plastic deformation of particles, and the work "X_1", which corresponds to the part of densification by rearrangement of particles (without plastic deformation of particles: for n=1). Also, the minimal compaction pressure "p_1", necessary for achieving a sufficiently strength compact, which is suitable for manipulation, can be determined.

In this study, the exactness of the compaction equations proposed by Heckel, equation (4), Kawakita, Lüdde, equation (6), Pamelli and Filho, equation (8) and Parilak et al., equation (14) was compared for four different powder systems. The employed equations were expressed in a linear form $f_1(P) = a_n \cdot f_2(p) + b_n$. Thus, a quantitative comparison can be made by linear regression analysis.

2. Experimental

Starting powders employed in the experiment were - Höganäs iron powders ASC 100.29, Höganäs prealloyed powder ASTALOY 85 Mo, electrolytic copper (sieved to 40 μm), and natural graphite CF 4 (sieved to 40 μm). HW wax as lubricant was added to all mixtures in the amount of 0.8 wt. %.

Four powder mixtures were prepared by homogenisation in the mixer Turbula. Formulation, chemical composition and basic properties of the analysed powder systems are given in Tab.1.

Table 1 Formulation, composition and basic properties of experimental powder mixtures

Code	**Formulation**	Composition, wt.%]	ρ_0 [1)] [g.cm^{-3}]	ρ_{th} [1)] [g.cm^{-3}]	**HMV 0.025**	**P_0** [%]
A	ASC 100.29	Fe+0.8HW	3.080	7.4042	128	58.40
A1	ASC100.29 +electolytic Cu+graphite	Fe+3Cu+0.7C +0.8HW	2.807	7.3415		61.77
M	Astaloy 0.85Mo	Fe+0.85Mo+0.8HW	3.033	7.4515	139	59.29
M1	Astaloy 0.85Mo +electolytic Cu+graphite	Fe+0.85Mo+3Cu +0.7C+0.8HW	2.717	7.3565		63.06

[1)] ρ_0 and ρ_{th} are the apparent and theoretical densities of the powders

Cylindrical specimens Ø10x10 mm^3 were compacted under the pressing pressures ranging from 50 MPa to 700 MPa. The green density was determined by weighting and dimensions measurement. The microhardness HVM 0.025 of particles (LECO…..) was measured on the metallographic cross-section of powder compacts.

3. Results and discussion

The microstructure of the compacts pressed at low pressures (up to 100 MPa) consisted of interconnected pores and particles without significant plastic deformation. At higher pressures, due to the plastic deformation of particles, the porosity is reduced. The plastic deformation of particles (strain hardening) is reflected in increasing microhardness of particles, Tab.2.

Table 2 Microhardness HVM 0.025 of powder particles in compacts pressed at 100 – 700 MPa

Pressure	100 MPa	200 MPa	400 MPa	600 MPa	700 MPa
Powder A	128	157	165	199	224
Powder M	139	154	167	184	194

The compaction equations (4), (6), (8) and (14) expressed in a form: $f_1(P) = a_n \cdot f_2(p) + b_n$ give straight-line relationships and a quantitative comparison of the exactness of the used compaction equations was possible by linear regression analyses. The values of the constants a_n and b_n, and the correlation coefficients r are given in Tab.3 and Tab.4.

Table 3 The constants a_n, b_n and correlation coefficients r for the dependence $f_1(P) = a_n \cdot f_2(p) + b_n$

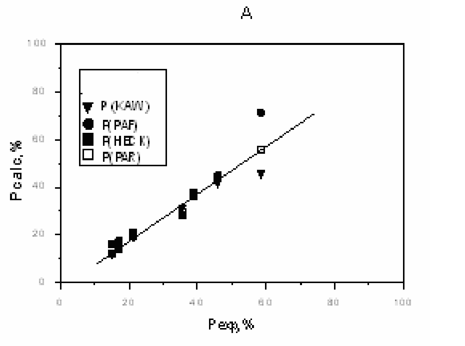
Powder	Equation (4)			Equation (6)		
	Constant a_2	Constant b_2	r	Constant a_3	Constant b_3	R
A	0.0018	0.7325	0.9888	127.108	1.857	0.9789
A1	0.0036	0.9380	0.9892	36.392	1.653	0.9627
M	0.0030	0.7947	0.9994	68.280	1.732	0.9786
M1	0.0036	0.7835	0.9883	47.648	1.591	0.9938

Table 4 The constants a_n, b_n and correlation coefficients r for the dependence $f_1(P) = a_n \cdot f_2(p) + b_n$

Powder	Equation (8)			Equation (13)		
	Constant a_4	Constant b_4	r	Constant a_7	Constant b_7	R
A	0.0622	0.2951	0.9899	0.6755	0.01686	0.9893
A1	0.1204	0.1039	0.9796	0.5958	0.05528	0.9865
M	0.1030	0.0819	0.9892	0.6714	0.02774	0.9940
M1	0.1172	0.0197	0.9849	0.6521	0.03694	0.9940

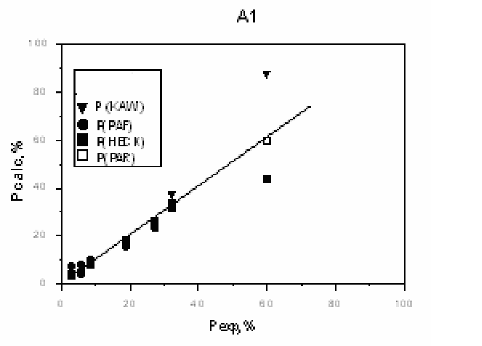
The values in Tab.3 and Tab.4 show that the correlation coefficients R are relative high for all used equations. The highest values of r were obtained for the equation (13) given by Parilak et.al. [10, 11]. The lower values of a_7 (eq. 13) for A1 and M1 than for A and M powders show that the addition of copper and graphite increases the compressibility of powders. Similarly, the lower values of the constants a2, a3, and a4 in the equations (4), (6) and (8) for A1 and M1 powders than for A and M powders, confirm the increasing of compressibility with copper and graphite additions.

In Figs.1-4 are presented the relationships between calculated and experimental determined porosities. The relationships between the Pcalc and Pexp are expressed by diagrams and correlation equations.



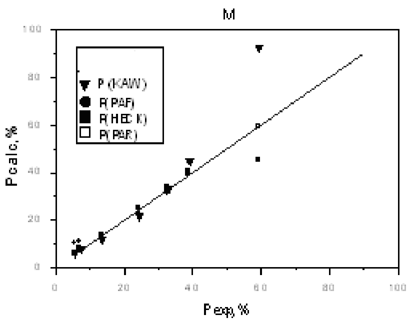
According to equation:
(4): $P_{calc} = 4.41 + 0.817\ P_{exp}$; r=0.9954
(6): $P_{calc} = 5.15 + 0.846.\ P_{exp}$; r=0.9791
(8): $P_{calc} = -6.75 + 1.270\ P_{exp}$; r=0.9752
(13): $P_{calc} = 0.012 + 0.995\ P_{exp}$; r=0.9954

Fig.1 The relation between P_{calc} and P_{exp} (powder A)



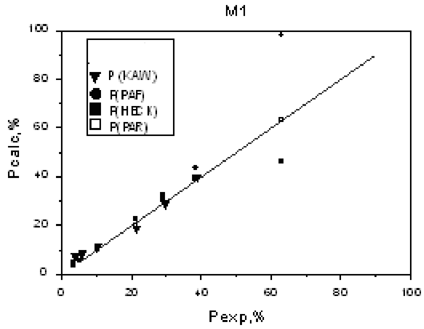
According to equation:
(4): $P_{calc} = 3.29 + 0.740.\ P_{exp}$; r=0.9759
(6): $P_{calc} = 3.42 + 0.821.\ P_{exp}$; r=0.9724
(8): $P_{calc} = -6.23 + 1.460\ P_{exp}$; r=0.9839
(13): $P_{calc} = -0.28 + 1.002.\ P_{exp}$; r=0.9974

Fig.2 The relation between P_{calc} and P_{exp} (powder A1)



According to equation:
(4): $P_{calc} = 3.59 + 0.790\ P_{exp}$; r=0.9744
(6): $P_{calc} = 4.02 + 0.840\ P_{exp}$; r=0.9815
(8): $P_{calc} = -8.70 + 1.521.\ P_{exp}$; r=0.9688
(13): $P_{calc} = -0.21 + 1.001.\ P_{exp}$; r=0.9982

Fig.3 The relation between P_{calc} and P_{exp} (powder M)



According to equation:
(4): $P_{calc} = 3.85 + 0.756.\ P_{exp}$; r=0.9645
(6): $P_{calc} = 2.04 + 0.909.\ P_{exp}$; r=0.9906
(8): $P_{calc} = -7.81 + 1.527\ P_{exp}$; r=0.9760
(13): $P_{calc} = -0.20 + 1.004.\ P_{exp}$; r=0.9992

Fig.4 The relation between P_{calc} and P_{exp} (powder M1)

The results given in Figs.1-4, show that the differences between the P_{exp} and P_{calc} are minimal in the range of middle values of porosity for all used equations. In the case of the equations (4), (6) and (8), relative high differences were identified in the area of the lowest and the highest porosity. In the case of the equation (13) P_{calc} and P_{exp} are practically equal for both - high and low porosity.

Based on the equation (12) [10, 11] the values of "p_1" (minimal compaction pressure necessary to obtain sufficiently strength compact), values of the "total work, X_n," (necessary to obtain full density by rearrangement and plastic deformation of particles), and values of the work "X_1" (necessary to obtain full density by particle rearrangement) were calculated. The values of p_1, X_n, and X_1 are listed in Table 5. The results showed that the pressure p1 is controlled by the iron powder type, while the values of X_n and X_1 depend on the composition of powder mixtures. Higher compressibility of the powders A1 and M1 than A and M is reflected in the lower values of X_n.

Table 5 The values of p1, X_n, and X_1 calculated from the equation (12)

Powder	Pressure p_1 [MPa]	X_n [%.MPa]	X_1 [%.MPa]
A	115	323	35
A1	117	121	11
M	121	163	32
M1	122	135	17

4. Conclusions

The compaction behaviour of four types of powder mixtures was analysed. The exactness of four equations proposed by Heckel [4], Kawakita [5], Filho [6], and Parilak et al. [10, 11] was quantitative tested. The best correlation of calculated and experimentally determined porosity exhibited the equation (12) given by Parilak et al. The compaction behaviour of the tested powders was characterised by the minimal compaction pressure p1, necessary to obtain sufficiently strength compact, by the "total work X_n", necessary to obtain full density by rearrangement and plastic deformation of particles, and the work "X_1", necessary to obtain full density by particle rearrangement.

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