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CHARACTERISATION OF POWDERS UP TO DATE LAWS OF COMPRESSION

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Summary :

Many studies have been carried out in order to be able to describe the relationship d = f(P) of the compression phase of a powder in a more or less empirical fashion. Among the different laws, that of Heckel is the more frequently applied. The inconvenience of this, however, is that it does not bear witness to the behaviour of the powder at extremely low pressures. Two new laws are described hereinafter (a complement to Heckel's Law and a new model), in attempt to solve this problem. The outcome of these equations forms the basis of the identification of the analysed powder, and its statistical sample testing contributes to product quality control.

Key words :

Characterisation – Powders – Compressibility – Statistical process control

1. Introduction :

Powder compression is a major part of powder metallurgy. This phase, which consists of a progressive reduction of porosity under the effect of a pressure field, brings complex phenomena into play.

Many studies (1-3) have been carried out in order to determine the equations which would allow to describe the density = f (pressure) in a more or less empirical fashion.

Thus, HECKEL's law, one of the more frequently used laws, describes the re-densification of a compressed item in the field of plastic deformation of particles. The main inconvenience of this Law is its inability to define the behaviour of a powder at the lowest possible pressures, which are those used in industrial processes of compression.

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This is the reason for which further research has been carried out on the basis of Heckel's Law in search of a new law to describe the relationship density = f (pressure) also for the lowest pressures.

2. Heckel's Law and its complement at low pressures :

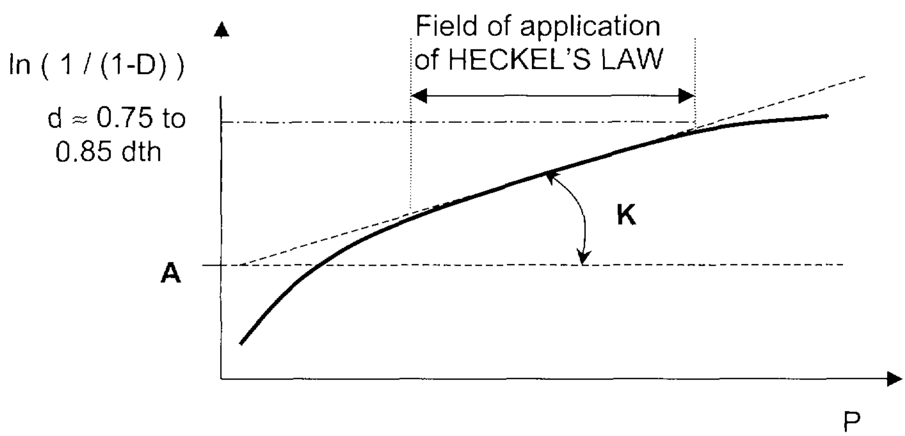
HECKEL suggests the following relationship :

$$\ln(1/(1-D)) = A + KP \quad ①$$

with: P = Pressure
d = density measured for a given P
dth = theoretical density of the non-porous material
D = d / dth
A, K constants, characteristic of the powder in question

By plotting ln(1/(1-D)) = f (P), in the adapted pressure field (200 to 1000 MPa for W powders for example), we get a straight line, of which the ordinate at origin A and the slope K characterise the powder (or the mix of powders) in question. (see figure 1).

Fig 1. Heckel's Law – Determination of parameters A and K



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In order to have data which also describes the behaviour of powders at the lowest pressure fields, CIME BOCUZE carried out studies intending to demonstrate the fact that the deviation between Heckel's Law and the actual density measured is a linear function of ln(P).

The suggested relationship is as follows:

$$\ln(1/(1-D)) = A + KP - (B + C\ln(P)) \qquad ②$$

Thus, Heckel's Law, and its complement at low pressures may be used to characterise metal powders or powder mixes. Parameters A, K, B and C would hence constitute the identification for the behaviour of the characterised powder under compression. Generally speaking:

- A and B are relative to the morphological and topological characteristics of the basic powder grains.

- K and C are representative of the energy to be dispersed to break the inter-crystalline bonds, mainly on agglomerate levels, and to plastically deform the inter-crystalline necks.

This compressibility test is used on a daily basis during powder acceptance inspection procedures before they are submitted to production lines.

For example, both table 1 and figure 2 specify the typical values of parameters A, K, B and C of Heckel's Law and its complement, for four different supply sources of tungsten 4-5 µm fisher powders.

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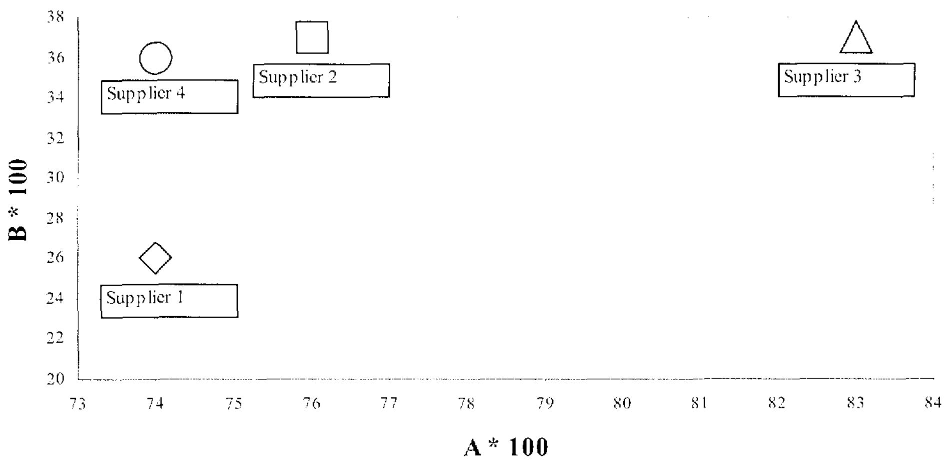
Tab 1. Parameters A, K ,B and C for different supply sources of tungsten 4-5 µm fisher powder

	Field 200 – 1000 MPa **ln (1 / (1-D) = A + KP**				Field 0 – 200 MPa **ln (1 / (1-D) = A + KP – (B + C ln P)**			
	A (* 100)		K (* 100 000)		B (* 100)		C (* 1000)	
	M	σ	M	σ	M	σ	M	σ
Supplier 1	74	(2.0)	59	(2.0)	26	(2.0)	46	(3.0)
Supplier 2	76	(3.0)	64	(1.0)	37	(2.0)	63	(3.5)
Supplier 3	83	(2.0)	60	(0.5)	37	(2.0)	63	(4.0)
Supplier 4	74	(1.5)	61	(1.5)	36	(2.0)	63	(4.5)

- M : mean
- σ : standard deviation

Fig 2. 4-5 micron tungsten powders - Heckel's Law parameters



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Thus, the following points are highlighted :

- At low Pressure: The "Supplier 1" powder differs from the other 3 due to its significantly weaker parameters B and C, to be linked with a lesser degree of agglomeration -aggregation owing to the characteristics of this supplier's production process.

- At high Pressure: The "Supplier 3" powder differs from the others due to its significantly higher parameter A. This is linked to a specific small grain/large grain size distribution favouring optimum packing at zero pressure.

- "Supplier 2" and "Supplier 4" powders, of which the morphologies are closely related, show very similar parameters A, K, B and C.

3. Relationship d = f(P) – new equation proposal :

Use of HECKEL's model and its complement at low pressures enables the characterisation and sample testing of the identification of compressed powders and powder mixes used in powder metallurgy.

The inconvenient aspect of this methodology is that two distinct mathematical models must be used in order to describe the behaviour of a powder throughout the entire pressure field in question.

Figure 3 shows experimental results d=f(P) for the 4 previously studied tungsten 4-5 µm powders.

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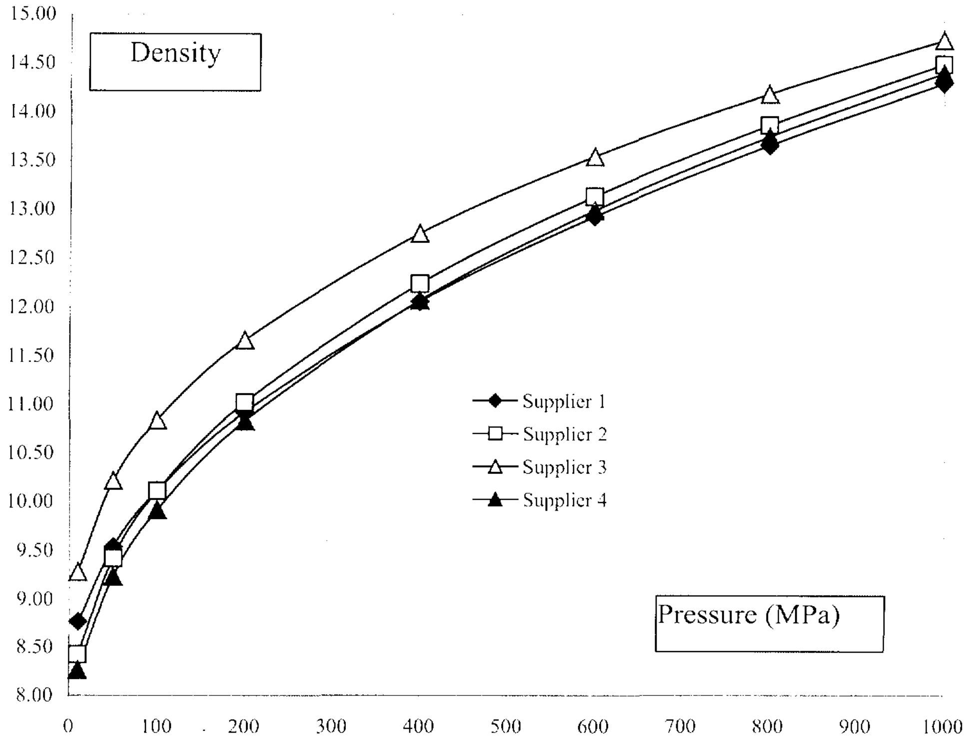
Fig 3. 4-5µm tungsten powders – density = f (pressure) – Experimental data



The curves obtained appear to be similar to "power law" curve results. It was therefore interesting to check whether it was possible to offer a model based on this principle which described the entire pressure field to be studied.

The new relationship offered is as follows:

$$\mathbf{(d\text{-}do) / (dth/do) = K\, P^n} \quad ③$$

where: P = Pressure
d = density measured for a given P
dth = theoretical density of the non-porous material
do, K, n = constants characteristic of the powder in question

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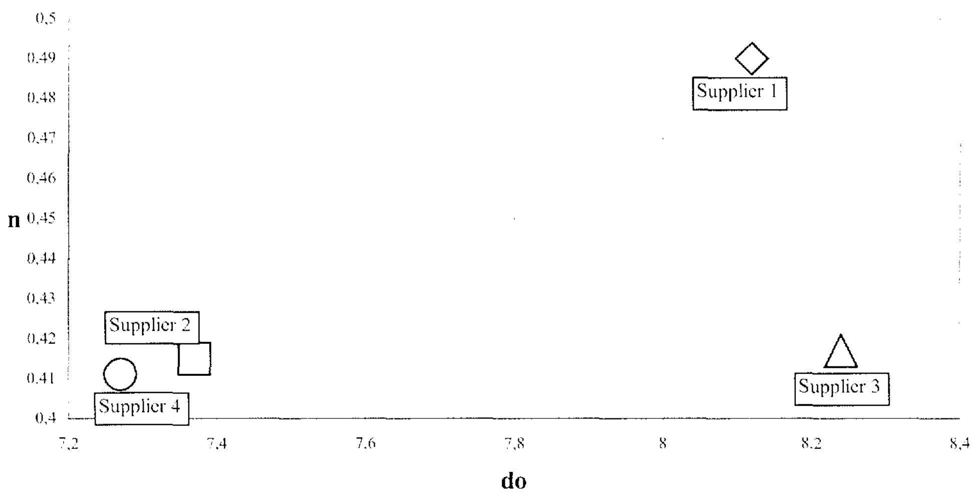
This relationship was applied to previously studied tungsten 4-5 µm powders in order to determine the parameters; do, K and n specific to these powders (see table 2 and figure 4). The correlation coefficient ($R^2 > 0.99$) suggests the adequacy of the empirical results to the law proposed.

Tab 2. Tungsten 4-5 µm powders
Parameters; do, K and n of the law **(d-do) / (dth/do) = K P^n**

	do	K	n
Supplier 1	8.12	0.0186	0.491
Supplier 2	7.37	0.0340	0.415
Supplier 3	8.24	0.0326	0.417
Supplier 4	7.27	0.0345	0.411

Fig 4. Tungsten 4-5 µm powders – parameters; do and n



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These results highlight the specific behaviour of the powders provided by each of the suppliers. In particular, they confirm the conclusions of the HECKEL test:

- Closely related behaviour of powders provided by suppliers 2 and 4.

- Specific behaviour of powders provided by suppliers 1 and 3.

Moreover, it was necessary to ensure the new model proposed could also describe the behaviour d = f (P) of a large spectrum of metal powders and powder mixes, and possibly even powders with an added organic binder.

Tests were therefore performed on the following samples:

- Submicronic tungsten (W) powders

- Molybdenum (Mo) powder

- Nickel (Ni) powders

- Silver (Ag) powders

- Mix of tungsten-nickel-iron (W-Ni-Fe) powders

- Granules of a mix of tungsten-nickel-copper (W-Ni-Cu) powders + organic binder

The results obtained are shown in table 3.

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Tab n°3. parameters; do, K and n of the law **(d-do) / (dth/do) = K P^n** for different types of powder

	do	K	n	R^2 correlation coefficient
Non-milled submicronic W powder	3.94	0.0744	0.274	0.997
Milled submicronic W powder	6.63	0.0188	0.445	0.997
Mo powder	1.83	0.1033	0.288	0.999
Ni powder	3.17	0.024	0.51	0.998
Ag powder	1.52	0.1559	0.283	0.998
W-Ni-Fe mix	8.05	0.0196	0.487	0.999
W-Ni-Cu granules + organic binder	8.23	0.1861	0.22	0.991

This table shows that regardless of the type of powder or powder mix studied, the correlation coefficient obtained between the model and the experimental data is very high for the density-pressure field in question.

This new law may thus be used for the acquisition of the identification of a standard powder as well as for the sample testing of a given supplier by means of Statistical Process Control (SPC) type sample testing of this powder's parameters do, K and n.

To give an example, the sample testing of do and n for the tungsten 4-5 µm powder provided by Supplier 4 is shown in figures 5 and 6.

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Fig 5. Supplier 4 –SPC sample testing of the do parameter

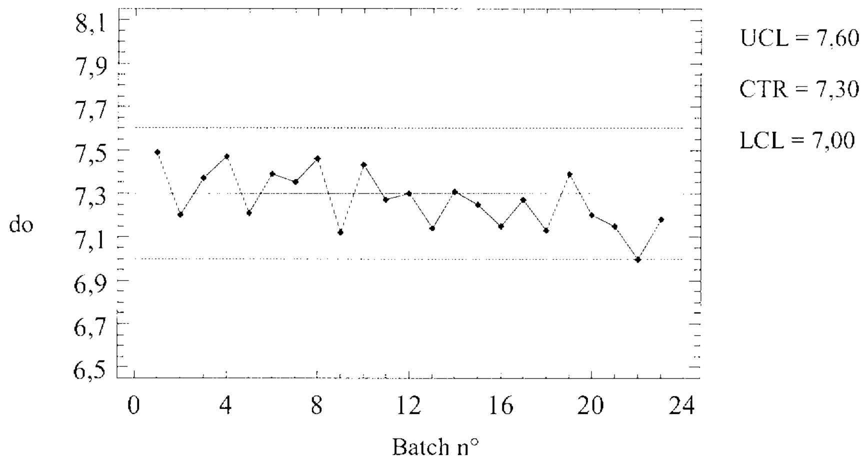
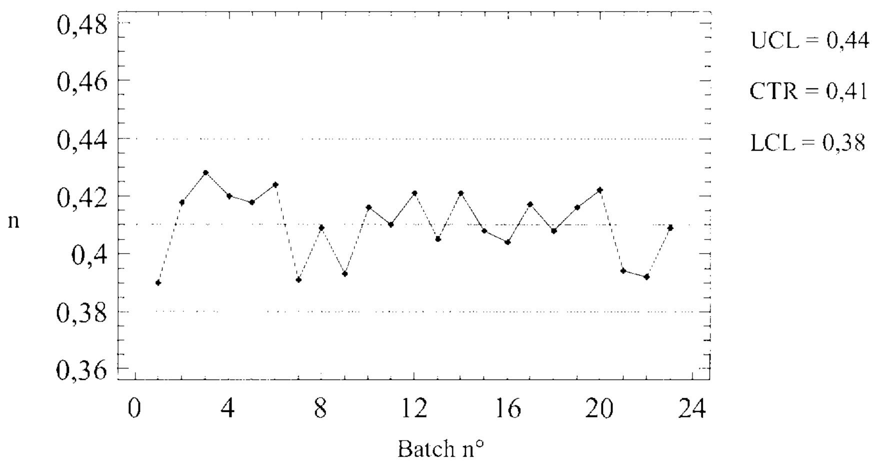


Fig 6. Supplier 4: SPC sample testing of the n parameter



- UCL : Upper Control Limit
- CTR : Central Line
- LCL : Lower Control Limit

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4. Conclusion :

Powder compression is a major part of powder metallurgy. It is therefore important to be able to acknowledge, or even model the behaviour of a powder or a mix of powders during this vital phase.

Among the different models offered, Heckel's Law is one of the more frequently used. It nevertheless shows the inconvenience of not being representative of powder behaviour for the lowest levels of pressure. This is why a complement to Heckel's Law at low pressures is suggested.

For each powder or powder mix, these two laws thus provide access to four parameters (A, K, B and C), which are characteristic of this powder mix. These 4 parameters form the basis of the powder's identification, and their sample testing may be used for quality control each time the powders are delivered.

It is, however, impractical to have to resort to two distinct laws in order to characterise a compressed sample for the entire pressure field to be studied. This is the reason for which a new model meeting this need has been suggested. The validity of this law was checked on a group of metal powders and powder mixes (which may have contained organic binders).

So, to describe the whole of the pressure field studied, it is thus possible to use only one model providing 3 parameters of the analysed sample's identification, of which the SPC would contribute to quality control procedures.

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