

fundamental aspects of the compaction of metal powders

E. Arzt, H. Fischmeister (*)

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The search for mathematical methods of predicting the increase in density of a metal powder during compaction has attracted numerous scientists, as it is both a technologically interesting and a scientifically challenging problem. This review outlines the way from simple curve fitting approaches to first attempts at constructing physical models which might form a rational basis for the improvement of powder compactibility or compaction procedures.

The models described range from considerations of the deformation of a single spherical pore to reasoning in terms of the deformation behaviour of spherical particles in regular and - more recently - random packings.

This paper will not deal with the way in which friction affects the distribution of pressure and density in a compact, nor with the effects of lubricants.

La recherche des méthodes mathématiques pour calculer la densification d'une poudre métallique pendant la compression a attiré de nombreux chercheurs, car elle est intéressante aussi bien pour la technique que pour la science. Cet exposé montre l'évolution des « curve fitting » aux premiers essais de création des modèles physiques qui pourraient devenir la base d'une amélioration de la compacité ou du processus de compression.

Les modèles décrits ici vont de l'idée selon laquelle la déformation d'un seul pore sphérique est représentative du comportement global d'un corps poreux aux investigations de la déformation des particules sphériques dans un empilement régulier et — plus récemment — désordonné.

Cette étude n'examine pas l'influence du frottement sur la distribution de la pression et de la densité dans l'échantillon, ni les effets de la lubrification.

INTRODUCTION

The compaction behaviour of powders is characterized by an increasing resistance to compression. Starting from the fill density D_0 of the powder in the die, densification proceeds rapidly at first, but continues at a steadily decreasing rate. Early in the history of powder metallurgy, it was accepted that the full or « theoretical » density of the powder

material could not be achieved in a single cold compaction step: the compression curves of all powders approach the theoretical density D_∞ in an asymptotic manner (fig. 1).

Attempts at a general description of powder compression have been of three kinds, which we shall review in turn:

— curve fitting with mathematical expressions designed for good adaptability but without regard to physical understanding of the process,

(*) Université des Mines, Leoben, Autriche.

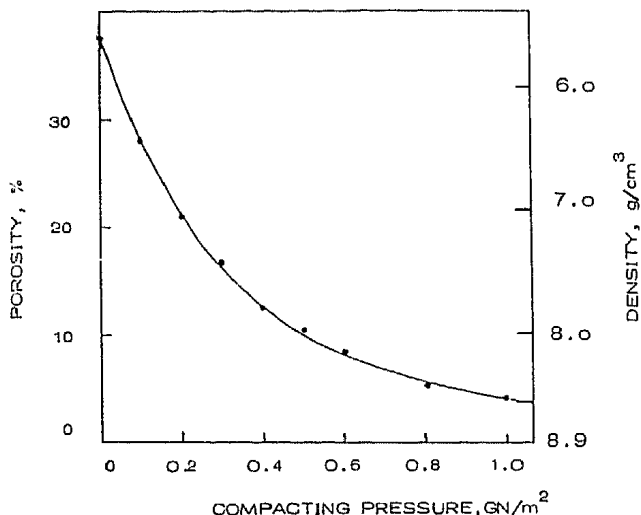


Figure 1. — Typical compaction curve (spherical bronze powder).

- physical models based on considerations of the pores in the powder,
- physical models based on the behaviour of the particles.

Ideally, the two last approaches should be identical, but in fact the necessary simplifying assumptions make them very different.

The degree of compression can be characterized either by the density achieved, $D(p)$, or by the remaining porosity, $U(p)$, both as a function of the pressure p . Recalling the relation

$$U = 1 - \frac{D}{D_{\infty}} \tag{1}$$

we will as a rule consider only porosity, i.e. the volume fraction of interparticle voids.

Comprehensive reviews of powder compaction have been published by Bockstiegel & Hewing (1) and by James (2).

CURVE FITTING

Walker (3) and Balshin (4) adopted a logarithmic expression of the following form :

$$\frac{1}{1 - U(p)} = - C_1 \ln p + C_2 \tag{2}$$

($C_1, C_2 =$ adjustable constants).

It is not surprising that this expression has a narrow range of agreement with experiment, since it cannot

reproduce the finite porosity U_0 before the onset of compression, nor the asymptotic approach to zero porosity. Correct behaviour at $p = 0$ and for $p \rightarrow \infty$ is obtained with an expression suggested independently by Konopicky (5) and Shapiro and Kolthoff (6) (out of context with powder metallurgy) :

$$U(p) = U_0 \exp(-C_3 p) \tag{3}$$

This expression reflects the intuitively appealing concept that compressibility at any moment is proportional to the remaining porosity :

$$-\frac{dU}{dp} = C_3 \cdot U \tag{4}$$

Considered in these terms, one would assume C_3 to be somehow related to the yield strength, σ_y , of the powder material. The first physical model to be suggested (Torre (7)) led to an identical expression, which also included a relation between C_3 and σ_y . It was based on the consideration of the shrinkage of a single spherical pore. This conceptual background lent some credibility to the KSK-formula [3], and its satisfactory agreement with experiment over the range of interest for technical pressing (at least when both constants are treated as adjustable parameters) has made it very popular.

When plotted in the form

$$\ln(1/U) = \ln(1/U_0) + C_3 \cdot p \tag{5}$$

as in figure 2, the KSK-relation predicts a linear plot whose slope is C_3 . Most compression curves, plotted in this way, exhibit such a linear portion at intermediate pressures, but at low and high pressures the KSK-relation underestimates the real porosity (fig. 2). This will be discussed further below.

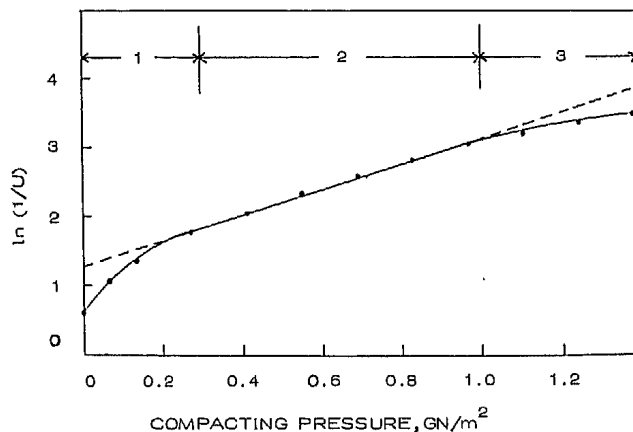


Figure 2. — Variation of $\ln(1/U)$ with compacting pressure for Atomet 28 iron powder (after Hewitt, Wallace and de Malherbe).

Kawakita (8) suggested a compaction equation which has proved applicable to many soft powders :

$$1 - \frac{D_0}{D} = C_4 \frac{C_5 \cdot p}{C_5 (p + 1)} \quad [6]$$

C_4 can be identified as the initial porosity U_0 , while C_5 , according to Brackpool (in James (2)) can be empirically correlated with the microhardness, HM , and the strain hardening behaviour of the powder material as characterized by the Meyer hardness-load-exponent γ

$$HM \cdot \gamma = C_6 \cdot \frac{C_4}{C_5 (1 - C_4)} \quad [7]$$

While these correlations suggest a physical background for the constants of the Kawakita formula, this has not been elucidated in terms of a model, and really satisfactory curve fitting requires both constants to be treated as adjustable parameters. Vdovic (9) suggested a still more adaptable formula :

$$U(p) = U_0 [(1 + C_7) p]^{-C_8} \quad [8]$$

In the authors' opinion, efforts to further improve curve fitting formulae are hardly justified. As will be shown below, powder compression is now recognized as a multi-stage process, with different mechanisms governing the various stages. In this light, the description of the whole compaction curve by a single expression is simply not meaningful.

THREE STAGES OF COMPRESSION

In 1946, Seelig & Wulff (10), later Donachie & Burr (11), Kunin & Yurchenko (12), and Heckel (13) suggested that three stages of compaction should be distinguished, as proposed by the ranges of invalidity of the KSK-formula (fig. 2). In the first stage, at low pressures, the reduction of porosity was ascribed to deformationless restacking of the particles. At intermediate pressures they postulated a second stage where the packing is stabilized and further compaction is possible only by deformation of particle contacts. In the final stage, at high pressures, bulk deformation was assumed to occur, the densification rate being determined by the strain hardening of the particle material.

Evidence for the restacking of particles has been summarized by James (2). The effect has been demonstrated, e.g., for beds of glass spheres, whose porosity could be reduced by 1.5 % without any fragmentation (Kakar & Chaklader (14)).

The occurrence of strain hardening has been demonstrated by Hewitt, Wallace & Malherbe (15) by means of X-ray line broadening measurements on

sections of compacts, and by Heliwell & James (16), who measured the microhardness of individual particles in polished sections of compacts produced with various pressures. Sundström & Fischmeister (17) emphasized the importance of « geometrical strain hardening », an increase in densification resistance which is due to the increase in contact area between the particles, and which they considered more dominant than ordinary « metallurgical » strain hardening.

One has to expect more or less extensive regimes of overlap between the three stages. In a study on particle sliding and deformation in a spherical bronze powder, Fischmeister, Arzt & Olsson (18) showed that at least for an easily deformable metal powder, restacking and contact deformation proceed concurrently until about half the initial porosity has been eliminated ; at this point, particle sliding has become insignificant for further densification. Despite these overlaps, restacking as a contributing mechanism of initial densification, and strain hardening (metallurgical and geometrical) as an obstacle to final densification must be accepted as essential parts of our understanding of powder compression.

CHANGES OF PORE GEOMETRY DURING COMPACTION

Torre (7) replaced the pore network by a single spherical pore surrounded by a rigid - perfectly plastic shell. Adopting the Tresca yield criterion, he calculated the pore volume as a function of hydrostatic pressure. The result is not only formally the same as the KSK-equation, it also predicts a value for the second constant :

$$C_3 = \frac{3}{2 \sigma_y} \quad \sigma_y = \text{yield stress} \quad [9]$$

However, comparison with experiment shows this to be too high for most powders. In fact, Heckel (13) established an empirical expression for this relationship :

$$C_3 = \frac{1}{3 \sigma_y} \quad [10]$$

This is about a fifth of the value predicted by Torre. Also, the Torre model has been criticized by Bockstiegel (1) on the grounds that the derivation neglects the compatibility equation. Bockstiegel (19) derived a threshold pressure $p_{\min}(r)$ below which plastic deformation of a shell with inner and outer radius r_i, r_o , is not achievable :

$$p_{\min} = \frac{2}{3} \sigma_y \frac{r_o^3 - r_i^3}{r_i^3} \quad [11]$$

This indicates that an isolated large pore should start to shrink at a lower stress than a smaller pore contained in an equal volume. Since it is based on Hooke's law, this reasoning applies only as long as plastic deformation does not actually occur. Furthermore, as pointed out by Sundström & Fischmeister (17), the extension of a single pore model to a multisize system is not trivial. E.g., in a regular array of pores of different sizes, adjacent pores must interact during the deformation to uphold the continuity of the system. Higher stress concentrations will develop at pores with small radii of curvature. This might well invalidate Bockstiegel's conclusion that larger pores should always yield first. Nevertheless, Bockstiegel's experiments showed that the larger pores do disappear preferentially from the distribution, which he determined by linear intercept measurements. The shape of the pore size distribution was found to remain unaltered throughout compaction; only the upper cut-off point is shifted to smaller pore sizes as compaction proceeds - an interesting observation that so far lacks a reasonable explanation.

Hewitt, Wallace & Malherbe (20) modified the Torre model to allow for strain hardening in the spherical shell. By numerical integration of the Prandtl-Reuss equations for a rigid-plastic material, they showed that even small amounts of strain hardening appreciable influence on the slope of the KSK- (or Torre-) plot, and they were able to obtain good agreement between calculated and measured slopes for an atomized iron powder and for aluminium powder. Sundström & Fischmeister (17) modelled the compression of a concave pore formed between four particles of initially circular cross section, using two-dimensional finite element calculations. They found that the pore kept its symmetry even under uniaxial compression. Despite the simplification from three to two dimensions, they obtained good agreement with experimental densification curves for iron powder when incorporating linear strain hardening ($d\sigma/d\varepsilon = E/200$), which is a fairly realistic approximation for the plastic behaviour of iron.

While the work of Hewitt, Wallace & Malherbe (20) demonstrated that the Torre model could not be made to reproduce the increased densification resistance in the third stage by including any degree of ordinary strain hardening (neither could it be explained by entrapped air), the model of Sundström & Fischmeister (17) gave satisfactory agreement with third stage behaviour thanks to the incorporation of geometrical strain hardening (the finite element calculations yield simultaneously the pore shape and pore size, i.e. porosity, for any given pressure, cf. fig. 3).

Cooper & Eaton (21) approached the problem of the gradual elimination of pores by two different mechanisms: larger pores are likely to be filled by restacking, while small pores can only be eliminated

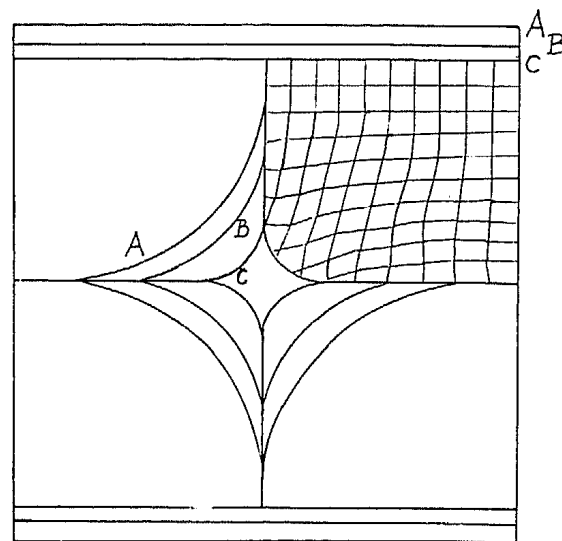


Figure 3. — Pore shapes at different porosities: $A = 13,7\%$, $B = 7,3\%$, $C = 1,6\%$ (after Sundström and Fischmeister).

by plastic deformation or fracture of the particles. They assumed intuitively that the probability of the disappearance of large pores would diminish exponentially with increasing pressure, and that a similar function with different parameters would describe the filling-in of the smaller pores:

$$U(p) = C_9 \exp(-C_{10}/p) + C_{11} \exp(-C_{12}/p) \quad [12]$$

However, neither the type of the function nor its constants can be stringently derived from physical principles, which brings this expression back to the level of a (unnecessarily flexible) curve-fitting formula.

Before leaving the topic of pore-governed compaction models, mention should be made of the continuum mechanical theories of compaction of presintered powder preforms (Kuhn et al. (22), (23), Green (24), Oyane et al. (25)), which concentrate on the relation between multiaxial deformation and densification, leaving the question of flow stress or pressure largely open.

MODELS BASED ON THE GEOMETRY OF PARTICLE CONTACTS

Recently, some researchers have turned their interest towards the deformation of particles in contact. To make the problem tractable, only spherical particles of uniform size are considered. This restriction is no longer unrealistic, since spherical powders are being processed technologically in

rapidly increasing quantities. The extension from monosize systems to powders with realistic size distributions should pose no principal problems.

A theory based on particle deformation could hope to encompass the second and third stages of compaction. At the beginning of stage II, a given particle has a certain number of nearest neighbours. As the external pressure increases, forces are transmitted into the powder mass which squeeze the particles together, widening the point contacts into contact areas. As compression proceeds, further neighbours are brought close enough to form additional contacts. At any given moment, the resistance to densification will be determined by the number and size distribution of the contact areas. It follows that the elements of a model for the compression of spherical powders must be:

- 1) The transmission of an externally applied pressure (uniaxial or isostatic) to the particles, to be expressed as a relation between applied pressure p and contact force f :

$$f = f(p).$$

At any given pressure, there will in fact be a distribution of contact forces, but for a simplified theory one could use the average contact force, \bar{f} .

- 2) The force carried by a given contact area is determined by the flow stress of the material and a constant of proportionality which depends on the deformation geometry:

$$f = C \cdot \sigma_f \cdot a \quad a = \text{contact area.}$$

- 3) The contact surface is related in a complicated manner to the residual porosity; this depends upon the change in particle shape when material is squeezed out from the contact zone (cf. fig. 3). If we know this change in particle geometry, we can establish the relation

$$a = a(U).$$

- 4) To conclude the treatment of stage II, we need to know the evolution of the number of actual particle contacts

$$Z = Z(U).$$

This will be determined by the original center-to-center distances in the particle packing, which can be described by the radial distribution function (RDF) of the packing. Such RDF's are known, e.g. for the atoms in a liquid, or for spheres randomly thrown into a box. For our purpose, it appears sufficient to know the RDF over a very narrow interval, e.g. from one to one-and-a-half particle diameters, and we hope that this could be covered by one single parameter.

- 5) Finally, to model the complete course of the compaction curve, we would have to know the contribution of particle restacking to initial densification. This could be expressed by making the RDF porosity-dependent.

The first problem, force transmission, has been considered by Molerus (26), who gives a stringent derivation of the transmission of pressure in a random sphere packing. Assuming an isotropic normal force distribution at the contact points, the contact forces can be regarded as equal for any orientation. It is now possible to sum up the contributions of the contacts to the propagation of stresses through an arbitrary plane. The result of this calculation is a relation between applied pressure and the average contact forces:

$$f = p \cdot \frac{4 R^2 \pi}{Z(1-U)} \quad [13]$$

This equation should be valid until the contact flats on the sphere surface impinge on each other. From then on, the normal contact force will not be the only component, and further densification will be due to a different stress distribution.

Another important contribution to the problem of force transmission is due to Davis & Deresiewicz (27). They used a discrete probabilistic model for predicting the compressibility of and the force distribution in a two-dimensional random packing. Even if their treatment is limited to the elastic range, the relation between force and the corresponding sphere compression being given by the Hertz equations, it is instructive to follow their reasoning and results.

Only normal forces are considered at the contacts, the packings are assumed to be in a geometrically stable configuration (no restacking). The problem is formulated as one of structural analysis; the sphere centers correspond to the nodes of the structural graph, and the contacts, to its branches. The authors state that a probabilistic model for generating an ensemble of dense random packings does not yet exist. Consequently, they transfer the randomness of particle distances to the matrix of contact stiffnesses. The random packing is thus replaced by a regular one whose stiffness moduli are produced by computer randomization, and whose distribution simulates the porosity, average coordination and internal-angle distribution of the actual sphere packing. The authors obtain distributions of contact forces ranging from zero to many times the average contact force; a significant traction of the contacts (about 20%) support no load at all.

The third problem, which concerns the relation of particle deformation to porosity, was first treated by Kakar & Chaklader (14). They considered regular packings of monosize spheres, such as the simple cubic, orthorhombic, body centered cubic, tetragonal, and rhombohedral packing.

They assumed the packing to be stable, maintaining its symmetry under a hydrostatic pressure (no restacking). Further they assumed that the material at the faces spreads evenly across the free surface of each sphere. The authors calculated the resulting increase in particle radius R as a function of the contact area, a , from the condition of constant volume. It turns out that R increases very slowly until the contact radius comes to equal on quarter of the particle radius; this happens at a porosity of the order of 30 %. (The authors limited their considerations to the porosity range above this point.) — The porosity is equal to the volume fraction of unfilled space in a unit cell of the sphere packing :

$$U(a) = U_0 - \frac{3}{2} (1 - U_0) \cdot \frac{a}{\pi R^2} \quad [14]$$

where U_0 depends on the packing type.

Experiments with lead and hot-compacted sapphire spheres agreed fairly well with the prediction for the orthorhombic packing, except for a faster decrease of porosity which was attributed to the gradual increase of coordination number during compaction.

In 1968, Kakar & Chaklader (28) combined their geometrical formula with a consideration of the contact forces, touching upon the second of our problems. On the basis of theoretical work by Hencky (29) and Ishlinsky (30) and of experiments by Tabor (31), they assumed the force for plastic yielding to be related to the contact area in the same manner as for a Brinell hardness impression :

$$f = 3 \cdot \sigma_y \cdot a \quad [15]$$

This holds as long as the deformed zones are small compared to the particle size, and strain hardening is negligible. The authors observed that the radius of the contact flats between the particles and the punch surface was approximately twice the average contact radius inside the compact. A balance of forces then yields

$$p = 7.2 \sigma_y \cdot \frac{a}{\pi R^2} \quad [16]$$

and substituting in equation [14], they obtain a porosity-pressure relation

$$U = U_0 - \frac{1 - U_0}{4.8 \cdot \sigma_y} \cdot p \quad [17]$$

This formula is only valid in the limited range considered by Kakar & Chaklader, i.e. for porosities above 30 percent. Good agreement is reported with experiments on spheres of lead, K-monel and sapphire.

The Kakar-Chaklader model is only a first indication of how problems 2 and 3 in our list could be solved. It has two severe weaknesses: first, the limitation to very early stages of compaction (where densification is probably governed by restacking rather than by particle deformation, so that the agreement between calculations and experiments must in fact be held to be fortuitous); and second, the assumption that the material squeezed out from the contact zone is evenly distributed across the remaining surface of each sphere. It seems probable that the displaced material is preferentially deposited in the vicinity of the contact zone. The description of the deformation of sphere-to-sphere contacts will have to be tested experimentally, and a refined mathematical treatment may prove necessary.

This still leaves the fourth problem in our list, a description of the change in coordination number. Our own experiments as well as those of other authors indicate that the so-called « random dense packing », which is a popular model for mono-atomic fluids, reproduces very closely the porosity and average coordination number ($Z = 7$) observed in packings of spherical powder particles. Its RDF, in the range of interest, can be approximated by a straight line. This allows a simple description of the increase in coordination as more and more neighbouring spheres are brought to touch the « representative » particle. Based on this simplification of the RDF, our own calculations were made to take this gradual change of coordination into account, leading to a function of contact area vs. porosity, which is in very good agreement with measurements (« random model » in fig. 4). Thus the Kakar-Chaklader solution of problem 3 can be carried a step further.

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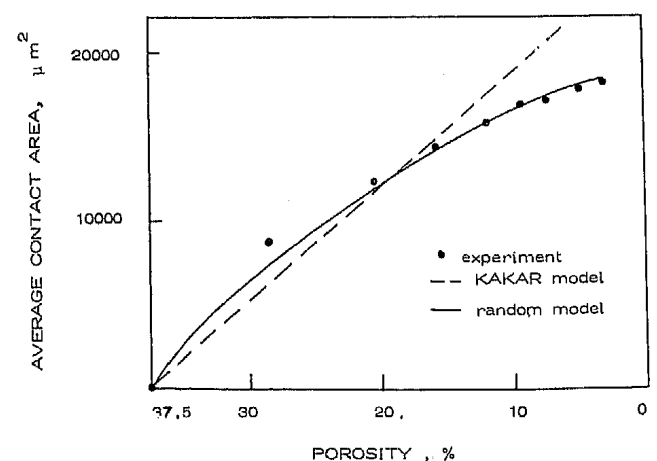


Figure 4. — Comparison between experimental values and theoretical predictions of average contact area vs. porosity.

of the change in coordination towards the end of compaction is that the third stage seems to be characterized by severe deformation of a few particles which are squeezed into the remaining gaps of unfilled coordination shells. This could explain the strong increase in densification resistance as full density is approached.

Acknowledgement

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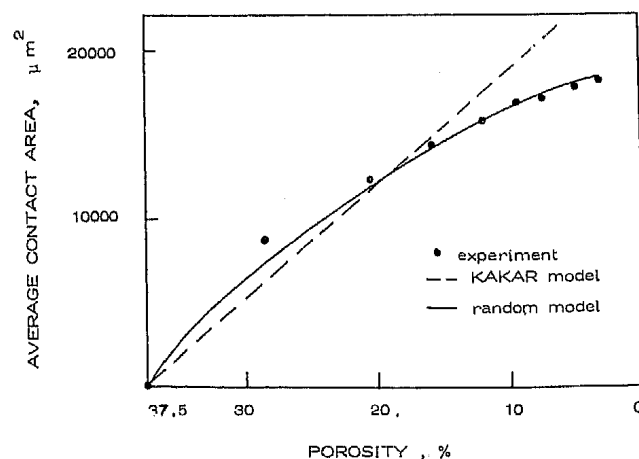


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discussion

M. ACCARY (Université de Clermont-Ferrand II). — Nous avons eu l'occasion de constater que « l'approximation sphérique » donnait une description étonnamment bonne des phénomènes de frittage. Avez-vous eu l'occasion de juger de cette valeur dans vos expériences ?

M. FISCHMEISTER. — Nous pensons aussi que le modèle sphérique constitue une bonne approximation pour la compression de poudres non sphériques. Mais, malgré les nombreuses indications sur la valeur du modèle sphérique pour des particules de poudres orientées au hasard, nous devons être très prudents jusqu'à une meilleure compréhension du comportement des poudres irrégulières. Les travaux en cours sur la géométrie des contacts de poudres irrégulières, à la Chalmers University, peuvent nous aider à améliorer notre compréhension.

M. ACCARY. — Quel est le rôle des états de surface (oxydes superficiels) ?

M. FISCHMEISTER. — Des variations de l'état de surface (films d'oxydes) influent sur les possibilités de glissement des particules, en jouant sur le coefficient de frottement et sur les pressions de contact où le « soudage à

froid » met un point d'arrêt au mouvement des particules. Nous n'avons pas encore de résultats d'observation, mais il paraît facile d'étudier ce phénomène avec des méthodes semblables à celles que nous utilisons présentement. Nous prévoyons d'étudier une poudre « fraîche » à compressibilité en vrac constante — avec divers revêtements entraînant divers comportements de surface.

M. CHERMANT (ISMRA, Université de Caen). — Je voudrais ajouter un commentaire à l'exposé de M. Fischmeister : à l'heure actuelle il est très important, pour mieux comprendre les mécanismes qui interviennent au cours de la compression des poudres avant frittage, de pouvoir atteindre toute information relative au nombre de contacts, à la surface de contact, aux glissements aux interfaces... Certaines de ces informations peuvent être obtenues par analyse métallographique quantitative, mais sont longues et laborieuses (les méthodes présentées étant manuelles), et dans le cas des surfaces de contact, il est absolument nécessaire que la valeur moyenne mesurée soit réellement la valeur caractéristique du matériau. Il semble que maintenant de nouvelles méthodes automatiques, basées sur la morphologie mathématique, soient applicables.

