# Compaction of homogeneous (Mn, Zn)-ferrite potcores

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The compaction of thin-walled potcore compacts (height 10 mm, diameter 15 mm, wall thickness 1 mm) with a high homogeneity is analysed. High homogeneity implies that the density is uniform throughout the compact. In order to attain this goal, internal and external friction are minimized while the mode of pressing and pressing velocity are optimized. Internal friction refers to friction between the powder particles, which is influenced by the particle size distribution and additives. The use of five different additives is evaluated. External friction relates to the friction between the powder and the die wall, which is influenced by the relative hardness of the die wall and lubricants. The effects of three different coatings and a lubricant on friction with the die wall have been investigated. The homogeneity of compacts with complicated shapes is affected by the formation of large local density gradients, which lead to differential shrinkage during sintering. By characterizing the shape of sintered products, it is shown that the inhomogeneity in compacts is reduced by optimizing the pressing mode and the pressing velocity. This improves the variation of the diameter over the height of the sintered potcores from 3 to 1.0%.

#### 1. Introduction

Die compaction is the most widely applied technique for the consolidation of ceramic powders. The main advantages of die compaction are its speed and versatility: a wide range of shapes can be pressed into compacts at a relatively low cost. However, die compaction yields compacts which are often less homogeneous than those obtained with more elaborate and expensive techniques such as slip-casting. In this paper, measures to improve the homogeneity of (Mn,Zn)-ferrite compacts pressed in a die are investigated. The results are used to optimize the pressing of cup-shaped compacts known as potcores. Sintered potcores made from (Mn,Zn)-ferrite are widely applied to telecommunications. In order to reduce production costs, optimum processing is required.

The homogeneity can be improved by reducing the friction between the powder particles and between the powder and the die surfaces [1]. In this study, granulated (Mn,Zn)-ferrite is used in order to improve the flowability. The granulate is obtained by spray-drying a slurry of (Mn,Zn)-ferrite particles, water, dispersant and binder. The ferrite particle size distribution is fixed on account of requirements with respect to the reactivity during sintering. Besides homogeneity, the binder influences the strength of the compact. Sufficient compact strength is also a criterion in the selection of a suitable binder. The use of five different binders is investigated.

The friction between the granulate and the die wall is largely influenced by the relative hardness of the die wall and the presence of lubricants on the granule surface. The hardness of the hardened steel (type: N1019) can be enhanced by coating the die surface. The effect of three different coatings has been investigated. An example of a lubricant is Zn-stearate, which can be added to the granulate after spray-drying. Note that the addition of Zn-stearate implies that the friction between granules differs from the friction between the ferrite particles. Granulates containing four different percentages of Zn-stearate have been tested.

Granulates are characterized in terms of the granule shape, the granule size distribution and the bulk or filling density. The various granulates are pressed into shapes suitable for characterization of the compact strength and of the compaction behaviour. The compact strength is determined by subjecting ring-shaped compacts (diameters: inner 11 mm, outer 16 mm) to diametral compression while the compaction behaviour is reflected by the relation between the applied pressure and the density of the granulate in a cylindrical die (diameter 12 mm).

Given optimum granulate and die characteristics, the parameters governing the compaction of potcores are varied. Potcores require sequential or simultaneous pressing from two sides. Sequential pressing has been performed in three stages: first, the bottom is pressed with part of the total displacement imposed in



Figure 1 Overview of the granulation, compaction and sintering stages in the processing of (Mn, Zn)-ferrite potcores.

this direction (stage I). Subsequently, the rim is fully pressed (stage II) before the bottom is pressed again by imposing the remaining displacement in this direction (stage III). The ratio of the length of stage I and III has to be optimized. Using the same overall displacements, potcores have also been pressed from two sides simultaneously. The effect on the dimensions of the sintered product is then established. Similarly, the effect of the pressing velocity is investigated by comparing products obtained by sintering compacts which have been pressed with different but constant velocities. The sintering process parameters are held constant in order to reflect the homogeneity of the potcore after compaction. Fig. 1 shows the parameters influencing the process stages.

#### 2. Preparation of granulate

Granulates containing five different types of binder have been produced with spray-drying. Selected binders are:

- acrylic (denoted as acrylic I),
- acrylic (denoted as acrylic II),
- polyvinylalcohol (PVA),
- mixture of PVA and acrylic II,
- polyethylene oxide (PEOX)

The solids content of the slurry had to be optimized: a too high solids content produced clogging in the spray-drier whereas a too low solids content lead to doughnut-shaped granules. Note that more water has to be evaporated when the solids content is low, which is reflected in the production costs. Spherical granules were obtained with the following slurry properties and spray-drying conditions:

Γ	A	BL	νE	I	Chara	cteriza	tion	of	granulate	e
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Binder type	Content (wt%)	Average size <sup>a</sup> (µm)	Bulk density (g cm <sup>-3</sup> )
Acrylic I	1.25	119	1.40
Acrylic II	0.86	180	1.52
PVA	1.00	182	1.40
PVA/Acrylic II	0.72	205	1.52
PEOX	1.50	70	1.40

<sup>a</sup> The variation in the diameter was constant and equals 1.35

- solids content between 50 and 70%
- addition of a dispersant to ensure wetting of the ferrite particles
- mass flow: laboratory  $\approx 4 \text{ kg h}^{-1}$ , industrial practice 500-1400 kg h<sup>-1</sup>

The average granule diameter was determined by laser diffraction. Furthermore, the bulk or filling density was determined using a measuring cylinder (diameter: 30 mm). Results are shown in Table I.

The binder content differs on account of differences in the evaporation and burnout characteristics of the binders during sintering. Note that the binder content should be as low as possible because (a) the binder has to be removed during the initial stages of the sintering process, (b) relaxation of the compact increases with an increasing binder content and (c) the compaction behaviour is affected if the bonding between the particles is too strong.

Given spherical granules, the bulk density is influenced by the granule size distribution and the density of the granules. In order to establish the effect of the granule size distribution on the bulk density, acrylic II granulate has been sieved to yield three fractions. The size ranges were  $106-150 \,\mu\text{m}$ ,  $212-250 \,\mu\text{m}$  and  $355-425 \,\mu\text{m}$ . The bulk density of these fractions was determined as well as that of various combinations of these fractions. It was found that the bulk density was approximately constant and equal to the bulk density of the original granulate. Consequently, the variation of the granule size is too small to produce a denser packing. Differences between the bulk densities can therefore be attributed to differences in the granule density can be correlated to differences in the solids content of the slurry, which is consistent with literature [2].

#### 3. Compact strength

#### 3.1. Significance of strength

The strength of a compact can be defined as the force required to produce failure normalized with the compact area on which the force is applied. Failure occurs when the particle packing in a certain region of the compact is sufficiently distorted. Local differences in the force experienced by the particles are due to (a) the manner in which the force is applied on the compact, (b) the density (distribution) within the compact and (c) the type and content of binder in the compact [3].

In order to establish the influence of the density and the binder on the strength, compacts of similar shape and size have to be subjected to similar loading conditions. Selection of the loading conditions depends on the significance of the strength; after ejection from the die, the compact is not necessarily loaded to reflect the minimum strength. With simple potcores, three types of strength can be distinguished: the axial strength, the radial strength of the rim and the radial strength of the bottom. After ejection from the die, a potcore is handled by the rim. Therefore, the radial strength of the rim has the greatest practical significance. This strength was measured by subjecting a ring-shaped compact to diametral compression.

During diametral compression, the compact is subject to tensile and shearing stresses [4]. Both stresses produce rearrangement of the particles within the compact. The process of rearrangement is governed by the particle size and shape distribution, the pore size distribution and the binder type and content. The influence of the binder can be modelled by considering two particles bonded by binder. Three situations can be distinguished: compression, shear and tension (Fig. 2).

During compression, the forces associated with friction between the particles are much larger than the strength of the binder. Consequently, the binder is smeared between the particles, which increases the strength. With shear and tension, the properties of the binder are more significant. However, with increasing density, or decreasing distance between the particles, the effect of friction between the particles becomes apparent. Therefore the effect of binder and adhesion to the particles on the strength is only observed when the bond is subjected to tension. Note that the adhesion of the binder to the particles is strongly influenced by the dispersant, which must be chemically compatible.



Figure 2 Schematic representation of the effect of particle motion on the bonds formed by binder molecules. The combined effect of the strength of the binder and adhension to the particle surface is only observed in the case of tension.

During diametral compression of a compact failure was often, but not always, initiated in the axial crosssectional plane in the centre of the compact. In other words, failure is not only a result of the circumferential tensile stress [5]. This indicates that the strength measured not only reflects the binder properties but also the particle shape and size distribution as well as the pore size distribution. The latter is determined by the compact density: the higher the density, the smaller the pore size and the narrower the pore size distribution.

When bending a simply-supported narrow rectangular compact, failure is initiated at the outer surface on account of tensile stresses. Although propagation of failure is also influenced by the compact density, the force at the onset of failure can be used as a measure for the strength of the bond between the particles [6,7]. Therefore, the selection of a binder based on the strength measured with diametral compression is not necessarily valid for other particle and shape distributions and compact densities.

#### 3.2. Diametral compression of compacts

Ring-shaped compacts (dimensions: inner diameter 11 mm, outer diameter 16 mm, height 10 mm) are pressed from each granulate to densities of 2.7 and  $3.0 \text{ g cm}^{-3}$  using a semi-automatic hydraulic Müller press (type TC 6, 3.11.15). At these densities, compacts with sufficient strength should be obtained. In general, pressing to higher densities requires excessively high pressures and promotes die wear.

The strength is related to the force required to crush compacts positioned sideways. The crushing process is carried out with a Zwick type 1435 tensile strength tester at a constant velocity of 20 mm min<sup>-1</sup>. The strength S is calculated from [8]

$$S = 6F(d_{o} + d_{i})/(\pi(d_{o} - d_{i})^{2}h)$$
(1)

where F is the maximum force,  $d_0$  the outer diameter,  $d_i$  the inner diameter and h the height of the compact. Note that deformation of the compact prior to compaction is not taken into account.

When crushing five compacts produced from each type of granulate, the variation was smaller than 3.0%

TABLE II Influence of binder type on the compact strength

Binder type	Strength S (MPa) Density = $2.7 \mathrm{g  cm}^{-3}$	Strength S (MPa) Density = $3.0 \text{ g cm}^{-3}$	
Acrylic I	1.38	2.34	
Acrylic II	1.18	2.14	
PVÅ	0.72	1.02	
PVA/acrylic II	0.41	1.28	
PEOX	0.14	0.68	

TABLE III Influence of binder content on the compact strength. Binder: acrylic I

Binder content (wt%)	Strength S (MPa) Density = $2.7 \text{ g cm}^{-3}$	Strength S (MPa) Density = $3.0 \text{ g cm}^{-3}$
1.00	1.10	1.97
1.25	1.38	2.34
1.70	1.75	2.87

in all cases. Table II shows the influence of the binder on the strength for the two compact densities.

Although the strength S increases with the compact density, it is also strongly influenced by the type of binder. In particular, the use of PEOX leads to unacceptably low levels of green strength.

The differences in strength are also influenced by differences in the binder content (Table I). Therefore, granulates containing 1.0 and 1.7 wt% of acrylic I binder have been produced. The variation of S with the binder content is given in Table III. The binder has a significant effect on the strength although the relative influence decreases with increasing content. Consequently, the strength of granulate containing acrylic II and PVA/acrylic II will be higher when increasing the binder content to comparable values (> 1.0 wt%). However, in view of the binder burnout constraint, the binder contents given in Table I are maintained.

#### 3.3. On diametral compression testing

The strength S has been determined from diametral compression measurements without taking the displacement prior to failure into account. Whether this displacement is indeed negligible was investigated by determination of the radial strength of the bottom. For this purpose, tablet-shaped compacts (diameter 24.5 mm, height 4.5 mm) have been pressed from acrylic II granulate to densities of 2.77, 2.88 and  $3.13 \text{ g cm}^{-3}$  with a manual Fontijne press.

Diametral compression of five compacts was performed at a velocity of 12 mm min<sup>-1</sup> with Zwick type 1435 tensile strength tester. It is found that the displacement prior to failure decreases with increasing density: 0.48 (2.77 g cm<sup>-3</sup>), 0.41 (2.88 g cm<sup>-3</sup>) and 0.38mm (3.13 g cm<sup>-3</sup>). This constitutes between 1.5 and 2.0% of the original compact diameter. In order to assess the influence of this displacement on the strength, an alternative interpretation of the diametral compression test is developed. The strength S is defined as the force required to produce failure normalized with the area between the compact and the cross-head at failure. The area equals the product of



Figure 3 Diametral compression of a cylindrical compact.

the compact height h and the width d. Because d is not measured, it is related to the displacement of the cross-head (Fig. 3).

Using Fig. 3, d can be expressed in the initial radius of the compact  $(R_0)$  and the displacement of the crosshead  $(\Delta R)$ 

$$d = 2(2R_o\Delta R - \Delta R^2)^{1/2} \tag{2}$$

The strength S then follows from

$$S = F/2(2R_o\Delta R - \Delta R^2)^{1/2}h \qquad (3)$$

where h is the compact height. The strength S of cylindrical compacts was found to be 1.58, 2.41 and 4.50 MPa for densities of 2.77, 2.88 and 3.13 g cm<sup>-3</sup> respectively. These values are slightly higher, but in the same order of magnitude as the strength measured by crushing ring-shaped compacts. This justifies the use of Equation 1.

## 4. Compaction behaviour

#### 4.1. Compaction curves

Because high strength is required, the acrylic binders appear to be the most suitable. However, apart from the strength, this choice is influenced by the compaction behaviour. For example, the strength of compacts pressed from PVA/acrylic II granulate with a density of  $3.0 \text{ g cm}^{-3}$  is comparable to the strength of compacts pressed from acrylic II granulate with a density of only  $2.7 \text{ g cm}^{-3}$  (Table II). If the pressure required to obtain the respective compacts is similar, the PVA/acrylic II binder would be preferred; a higher density reduces the shrinkage requirement during sintering which reduces the effect of inhomogeneity.

As a first indication, the pressure required to press cylindrical compacts (diameter 15 mm) with a density of 2.75 g cm<sup>-3</sup> has been determined for each granulate. The granulate containing PEOX binder typically required 32 MPa whereas the other granulates



Figure 4 Compaction curves for different granulates. Binder types acrylic ( + ), acrylic II ( $\bigcirc$ ), PVA ( $\triangle$ ) and PVA/acrylic II ( $\diamondsuit$ ).

required 90–95 MPa. Therefore, the PEOX binder seems to be an ideal choice. However, compacts with this binder have a relatively low strength. At high densities ( $> 3.3 \text{ g cm}^{-3}$ ), the increase in strength becomes negligible on account of increasing relaxation, which leads to reduction of the density and introduces cracks in the compact. Consequently, the choice of PEOX as a binder is not considered further.

The compaction behaviour of the other granulates has been characterized by measuring the compaction curves, which represent the relation between the applied pressure and the density of the granulate [9, 10]. In order to measure the compaction behaviour at low densities, the density is determined from the displacement of the punch rather than from the compact dimensions.

Compaction curves were measured by pressing granulate in a die (diameter 12.0 mm) with a manual Federhoff press. At 30 pressures, the displacement of the punch was recorded after allowing the displacement to reach a stable value. The measured curves are shown in Fig. 4.

Fig. 4 shows that the effect of the binder on the compaction curve is limited. Consequently, the choice of granulate containing acrylic I binder on account of the compact strength is the most appropriate.

## 4.2. Relation between the strength and the compaction behaviour

The compaction curves shown in Fig. 4 can be characterized with a three-stage model. The initial increase in the density is a result of rearrangement of the granules. This process is reflected in the non-linear section of the compaction curve at low pressures. The remainder of the compaction curve can be characterized by two linear correlations and a transition density. The correlations are defined as follows

$$\rho = A \log(P) + B$$
 when  $\rho < \rho_t$  (4a)

Binder type	$A (g cm^{-3})$	B (g cm <sup>-3</sup> )	$\rho_t$ (g cm <sup>-3</sup> )	C (g cm <sup>-3</sup> )	D (g cm <sup>-3</sup> )
Acrylic I	0.50	1.13	2.34	0.66	0.76
Acrylic II	0.51	1.16	2.48	0.69	0.70
PVÅ	0.58	0.99	2.34	0.69	0.73
PVA/Acrylic II	0.52	1.18	2.45	0.59	1.02

and

$$\rho = C \log(P) + D$$
 when  $\rho > \rho_t$  (4b)

where  $\rho$  is the density, *P* the pressure and  $\rho_t$  the transition density. The values for the parameters *A*, *B*, *C* and *D* are given in Table IV. Comparison of Tables I and IV indicates that differences in the granule density are reflected in the transition density. A higher granule density implies that the packing of the particles within the granules is denser. This increases the resistance of the granules to deformation and compression which results in filling of the large intergranular pores at a higher density.

Because the largest pores present determine the strength of the compact, the transition density  $\rho_t$  could be related to the compact strength. More precisely, the granulate has only acquired sufficient strength after the transition density has been exceeded. This can be expressed as follows

$$S = 0$$
 when  $\rho < \rho_t$  (5a)

and

$$S = k \log(P)$$
 when  $\rho > \rho_t$  (5b)

where k is a constant. Note that the logarithm of the pressure is taken because the strength generally varies linearly with the density at higher densities. The value of k can be determined from the constants A, B, C and D [11]. It is found that an indication of k is obtained from the ratio of the slopes C and A. For compacts containing acrylic I or acrylic II, this ratio is larger than 1.3, while it is smaller than 1.2 for compacts containing the other two binders. This is consistent with the strength determined by diametral compression of compacts.

The variation of k with the type of binder indicates that, above the transition density, the relation between the strength and the density varies with the binder. This implies that, at a density larger than the transition densities, the strength of compacts containing acrylic II binder will overtake the strength of compacts containing PVA binder. In view of the lower transition density and high value of k, the acrylic I binder is selected for the pressing of potcores.

# 5. Friction between the granulate and the die wall

Friction between the granulate and the die wall can be characterized in terms of the dynamic wall friction coefficient  $\mu$ , which is defined as follows [12]

$$\mu = F_{\mathbf{z}, \mathbf{w}} / F_{\mathbf{r}, \mathbf{w}} \tag{6}$$

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Figure 5 Die-wall friction measurement. During a measurement, preconsolidated granulate is pressed against the central plate (top). In this study, a compaction pressure of 220.0 MPa is used, while the test pressure amounts to 19.2 MPa. The force required to move the central plate downwards (velocity 6 mm min<sup>-1</sup>) reaches a stable value which is used for the determination of the dynamic wall friction coefficient (bottom).

where  $F_{z,w}$  is the axial force required to move granulate along the die wall surface and  $F_{r,w}$  is the radial force exercised by the granulate on the die wall. Note that the static wall friction coefficient  $\mu_s$ , which is defined with respect to the *initial* force required to move the granulate, is slightly larger than the dynamic wall friction coefficient. The value of  $\mu$  is determined from the data measured with the experimental setup (Fig. 5).

The central plate is produced from hardened steel (type: N1019) which corresponds to the material from which dies can be manufactured. A second plate was coated with a layer of W: C-H (thickness  $3-5 \mu m$ ) by physical vapour deposition [13]. The wall friction coefficient  $\mu$  was determined using both the uncoated and coated plate for four of the granulates. The average values obtained from three measurements are given in Table V.

Comparison of the wall friction coefficients in the uncoated die shows that granulate containing PEOX

TABLE V Wall friction coefficients for hardened steel plates

Binder	N1019	N1019, W:C-H
type	μ(-)	$\mu(-)$
Acrylic I	0.47	0.20
PVA	0.50	0.20
PVA/Acrylic II	0.42	0.17
PEOX	0.27	0.18

TABLE VI Wall friction coefficients for hardened steel plates. Binder type:acrylic I

Zn-stearate content (wt%)	N1019 μ(-)	N1019–W:C–H μ( – )	
0	0.47	0.26	
0.05	0.45	0.21	
0.2	0.30	0.18	
1	0.10	0.14	
2	0.09	0.11	

has a significant advantage over the other binders: this binder lowers both internal and external friction.

In order to verify whether the choice of W: C-H is optimal, two other coatings have been tested: plates were coated with TiN and TiC by chemical vapour deposition. The wall friction coefficients were found to be 0.40 for either case when using granulate containing acrylic I. Table V shows that this constitutes a significantly smaller improvement than obtained with a coating of W: C-H.

As well as coating the central plate, the die wall friction coefficient can be influenced by adding a lubricant (Zn-stearate) to the granulate after spray-drying [14]. Four different concentrations of Zn-stearate have been tested on plates with and without a coating of W: C-H (Table VI).

Table VI shows that large additions of Zn-stearate have the same effect as coating the die wall with W: C-H. This could be due to the formation of a layer of Zn-stearate on the surface of the plate. Unfortunately, high concentrations of Zn-stearate also tend to promote sticking of the granulate to the die wall. Furthermore, the concentration should be as low as possible because Zn-stearate has to be removed during the initial stages of the sintering process. Consequently, only small additions of Zn-stearate are feasible. Table VI shows that when the plate is coated with W: C-H and 0.05 wt% Zn-Stearate is added, the relatively largest reduction is observed; a further decrease of the wall friction coefficient  $\mu$  requires a strong increase in the Zn-stearate content. The effect of these modifications on the homogeneity of the potcore will be investigated in future studies.

#### 6. Pressing of potcores

#### 6.1. Standard conditions

Standard potcore compacts have been pressed from granulate containing acrylic I binder with a hydraulic press (TNO-IPL) which allows independent displacement of the lower and upper punch. In this way, displacement-controlled double-sided pressing of



Figure 6 Dimensions of potcore compacts.

potcores has been carried out. The dimensions of the potcore compact are shown in Fig. 6.

Given the final dimensions of the compact, the total filling height (FHT) and the filling height of the bottom (FHB) determine the density of the potcore compact. In practice, a density of approximately  $2.8 \text{ g cm}^{-3}$  is required to obtain a potcore with sufficient strength. Consequently, an FHT of 22.0 mm and an FHB of 2.1 mm are initially selected.

The potcores have been pressed in three stages: first the bottom is partially pressed (stage I), then the rim is fully pressed (stage II) and finally the bottom is pressed again (stage III). Inhomogeneity in the compacts is reflected in differences in the local shrinkage of potcore after sintering. Therefore, the potcore compacts have been sintered in an atmosphere-controlled kiln at a top temperature of 1350 °C. Subsequently, the sintered potcores have been characterized by measuring three diameters with a Heidenhahn extensometer: at either end of the potcore and in between where the diameter is smallest.

With these diameters, the inhomogeneity in the compact can be characterized in terms of the difference between the minimum and maximum diameter. An alternative is to consider the standard deviation in the average diameter. Using the latter, the inhomogeneity  $\zeta$  can be defined as follows

$$\zeta = 100\sigma_{\rm D}/D_{\rm av} \tag{7}$$

where  $\sigma_D$  is the standard deviation in the diameter and  $D_{av}$  the average diameter.

For the standard potcores, the effect of the relative length of stages I and III (I/III ratio) and the pressing elocity has been established by sintering five potcores pressed with each combination of parameters.

Table VII shows that a relatively long first stage (length I/III = 3:1) is disadvantageous for both velocities. Apparently, a density gradient is formed at the base of the rim. On account of the high external friction, this aggravates the axial density gradient in the rim. Comparison of the diameters confirms that the density at the edge of the rim is too high.

#### 6.2. Optimization of the homogeneity

Sintered potcores can be classified according to shape as shown in Fig. 7. The shape of the potcore is directly related to the compaction conditions. The latter refers to the pressing cycle, the pressing velocity and the

TABLE VII Inhomogeneity as determined from sintered potcores. Binder:acrylic I, FHT:22.0 mm, FHB:2.1 mm

I/III ratio ( – )	Velocity $(mm s^{-1})$	Inhomogeneity (%)
1:3		2.6
	10	2.5
1:1	1	2.7
	10	2.2
3:1	1	3.9
	10	4.1

magnitude of internal and external friction. Because of external and internal friction, an axial density gradient is expected in the rim of the potcore. This implies that the diameter of the rim at the edge is always larger than the diameter of the rim at some point towards the bottom. Consequently, cases 1A, 2B and 2C are unlikely to occur in practice. Only a very high pressing velocity combined with negligible external and internal friction could lead to these cases through movement of granulate towards the middle of the rim.

The relative density of the rim and the bottom determines whether case 1 or case 2 is observed. This is strongly influenced by the ratio of FHT and FHB. However, also the I/III ratio and the pressing velocity affect the movement of granulate into or out of the bottom. A long initial pressing stage produces movement of granulate from the bottom into the rim. However, at the same time, this restricts movement of granulate from the rim into the bottom during the second pressing stage. In this case, the axial density gradient in the rim is determined by the effects of internal and external friction.

With the standard potcores presented in Section 6.1, case 1B is observed for a low I/III ratio (1:3) irrespective of the velocity. Case 1C is observed for the other combinations of the I/III ratio and pressing velocity. Therefore, reduction of external friction should improve the homogeneity. Consequently, the die wall was coated with W:C-H and 0.05% Zn-stearate was mixed with the granulate. Five potcores were pressed for each (selected) variation of the I/III ratio, pressing velocities, FHT and FHB. Table VIII shows the effect on the (in)homogeneity.

All shapes were of the type 1C with the exception of the potcores pressed with FHB of 2.2 mm and a I/III ratio of 1:2. The latter has the shape of 2C, indicating that the density of the bottom is too high. That this is not the case when the I/III ratio equals 2:1 indicates that granulate is moved into the rim.

In terms of the inhomogeneity  $\zeta$ , a significant improvement compared to the standard potcores (Table VII) is observed for all combinations of parameters. Irrespective of the pressing velocity, the relatively short initial stage (length I/III = 1:2) is advantageous. This was also observed for the (high friction) standard potcores.

The effect of density gradients in the compact on the shape of the sintered product is reduced by increasing the compact density. However, increasing the filling heights (FHT and FHB) was found to decrease the homogeneity. This indicates that the increase in the



Figure 7 Effect of inhomogeneity on the shape of the potcore. First the diameters at either end are compared (index 1 or 2). Subsequently, the relative position of the intermediate extreme is classified (indices A, B, C).

TABLE VIII Inhomogeneity as determined from sintered potcores. Binder: acrylic I + 0.05 wt% Zn-stearate, coating: W:C-H

FHT (mm)	FHB (mm)	I/III ratio ( – )	Velocity (mms <sup>-1</sup> )	Inhomogeneity (%)
22.0	2.0	1:2 2:1	1 10	1.0 1.8
	2.2	1:2 2:1	10 1	1.4 1.5
24.0	2.0	1:2 2:1	10 1	1.2 2.0
	2.2	1: 2 2:1	1 10	1.4 2.1
24.0	2.2	1: 2 1:1 2:1	30 30 30	2.9 1.8 1.7

length of the pressing stages promotes the formation of density gradients. Therefore, an increase in the density should only be effected by increasing the density of the granules.

Table VIII shows that the pressing velocity has little influence on the homogeneity. This could be due to the relatively low velocities used. In order to establish the influence of the velocity, potcores were pressed at a velocity of  $30 \text{ mm s}^{-1}$ . The variation of the I/III ratio and the filling heights (FHT and FHB) are also given in Table VIII. Case 2C is observed when the I/III ratio equals 1:2 and 1:1 while case 1C is observed for a I/III ratio of 2:1. A short initial pressing stage (length I/III = 1:2) is not advantageous. Comparison of Tables VII and VIII shows that a higher pressing velocity requires an increase in the length of the initial pressing stage in order to prevent flow of granulate from the rim into the bottom. Overall, the increase in pressing velocity does not improve the homogeneity.

Further improvement of the homogeneity is pursued by modifying the mode of pressing: instead of sequential pressing, the potcore is pressed from two sides simultaneously. With simultaneous pressing, the velocity of the lower and upper punch are attuned so that the pressing from either side is completed at the same time. In order to compare with sequential pressing, potcores were pressed by simultaneous pressing using an FHT of 22.0 mm and an FHB of 2.0 mm.The rim was pressed with a velocity of  $10 \text{ mm s}^{-1}$  while the bottom was pressed with a velocity of  $0.8 \text{ mm s}^{-1}$ . The inhomogeneity based on five compacts was found to be 1.4%. This value lies between the inhomogeneity obtained when pressing sequentially for different I/III ratios and velocities. Consequently, simultaneous pressing does not produce further improvement of the homogeneity.

#### 7. Final remarks

When pressing thin-walled potcores, the compact strength has to be relatively high in order to allow handling. It has been shown that differences in the compact strength on account of the binder type can be quantified with the diametral compression test. The trends are reflected by the interpretation of measured compaction curves.

Optimization of the compaction of potcores has been performed in order to reduce the shrinkage differences in the sintered potcore. Methods employed are (a) reduction of the friction between the die wall and granulate and (b) suitable selection of the compaction conditions. The following optimum settings have been established

coating – W : C–H (3–5  $\mu$ m) lubricant – Zn-stearate (0.05 wt%) pressing mode – sequential pressing I/III ratio – 1:2 pressing velocity–1 mm s<sup>-1</sup>

With these settings, the inhomogeneity  $\zeta$  was improved from roughly 3 to 1.0%.

When increasing the pressing velocity to  $30 \text{ mm s}^{-1}$ , the I/III ratio has to be increased to prevent the formation of density gradients in the interface between the bottom and the rim. However, the inhomogeneity is always slightly larger.

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