

PROPERTIES AND COMPRESSIBILITY OF Fe/SiO₂ COMPOSITE COATED POWDERS

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Abstract

Soft magnetic composites prepared by powder technology consist of magnetically soft particles (usually Fe) which are coated with a suitable electro insulation layer. An important phase of their preparation is shaping without damaging the insulation layer. This contribution deals with the effect of both the size and shape of iron particles and the effect of binder addition (Silane) on the compressibility of composite coated powders Fe/SiO₂ prepared by the sol-gel method with a following calcination at 400°C. At low pressing pressures, the densification of coated powders is controlled by the shape of iron particles (the apparent density is higher for powders with spherical particles), while in the stage of prevalent densification by plastic deformation the compressibility depends on the ability of iron core to plastic deformation without any substantial effect of the SiO₂ coating. The compressibility was quantified by the compression ratio, relative density and parameters of a compaction equation.

Keywords: *composite coated powders Fe/SiO₂, compressibility, microstructure*

INTRODUCTION

Modern technologies and new powerful devices require new materials with exceptional properties that need specific approaches involving unusual chemical concepts and unconventional technologies. One specific group of materials which needs a spatial "network" microstructure are the soft magnetic composites consisting of magnetically soft particles (usually Fe) coated with a suitable electro insulating layer, such as ceramics or polymer, e.g. [1-3]. The development of these materials is motivated by the objective of replacing the conventional soft magnetic materials, i.e. laminated steel sheets by cheaper powder composite materials with comparable mechanical and electromagnetic properties, e.g. [4]. Currently, this is mainly the conception of modern materials, such as Fe/FePO₄ [5,6], Fe/MgO [7,8] and Fe/SiO₂ [9]. Some of them are at the research level, while others are industrially applied, e.g. [10,11]. Considerable attention is also given to materials based on Fe/MgO powders, e.g. [12] because of their higher thermal stability. Less information has been published about SMCs based on the Fe/SiO₂, which are interesting because of their favourable thermal stability and relatively simple material base. A critical step of the production technology is compaction, in which the highest possible density must be achieved without damaging the electro insulating coating. Different methods are used, but the most economically advantageous approach is cold pressing. This contribution focuses on the effect of the iron particle properties (shape and size) and the effect of the addition of Silane as a binder on the compressibility and microstructure of Fe/SiO₂ composite powders prepared by the sol-gel method.

EXPERIMENTAL PROCEDURE

The starting powder used was commercial water atomised iron powder ASC 100.29 (Höganäs AB) in as received state - with particles of irregular shape, Fig.1a (signed N) and after milling in a mill Pallmann REFL18 at 11,000 rpm thus obtaining particles of a spherical shape, Fig.1b (signed M). Milled powder was annealed at 600°C for 60 min in an atmosphere of $N_2+10\%H_2$. Three sieved fractions with different particle sizes were prepared for testing: <200 μm (for powder N, signed P) or <160 μm (for powder M, signed P), 100-160 μm (signed 1) and 45-63 μm (signed 2), Table 1.

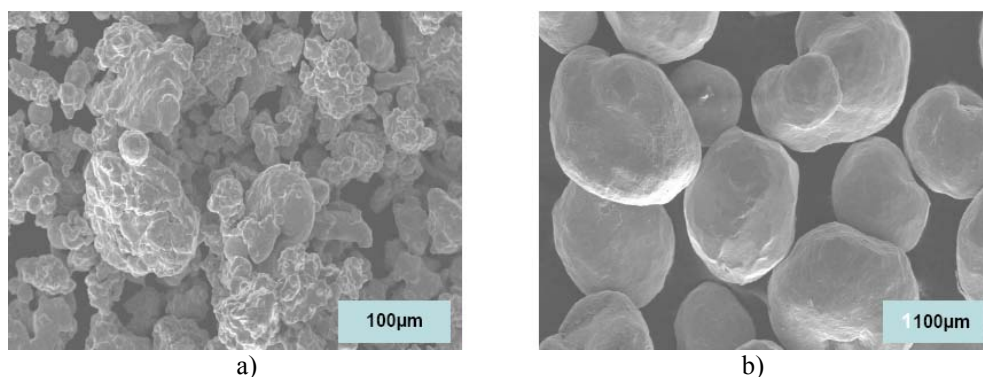


Fig.1. Iron powder particle shape: a) as received, b) after milling.

Tab.1. Fe/SiO₂ powders prepared from two iron powders with different particle shape.

| Coated Fe/SiO ₂ powders | | Iron powder | | Calcination |
|------------------------------------|-------------------------|--------------------|----------------|-----------------------------------|
| Designation | Coating cont. [wt.%] | Particle size [μm] | Particle shape | |
| NPB | 0.97 | <200 | irregular | at 400°C for 3 hours in air |
| N1B | 0.97 | 100-160 | | |
| N2B | 0.98 | 45-63 | | |
| MPB | 0.98 | <160 | spherical | |
| M1B | 0.98 | 100-160 | | |
| M2B | 0.99 | 45-63 | | |
| NPB+S | 0.97 | <200 | irregular | |
| N1B+S | 0.97 | 100-160 | | |
| N2B+S | 0.98 | 45-63 | | |
| MPB+S | 0.98 | <160 | spherical | |
| M1B+S | 0.98 | 100-160 | | |
| M2B+S | 0.99 | 45-63 | | |

The Fe/SiO₂ powders were prepared by sol-gel method according to Yoldas [13], as follows: 50 g iron powder + 1.5 ml H₂O, 7g Si(OR)₄ and 50 ml C₂H₅OH were mixed in a magnetic stirrer for 60 min composite coated powders Fe/SiO₂ at 25°C. Sol-gel coated powders were calcined at 400°C for 3 hours in air. Silane (3-glycidioxypropyltrimethoxysilane) was used as a binder added in an amount of 1.0 wt.%. Table1 summarizes the several experimental SiO₂ coated Fe composite powders prepared from iron powders of different particle shape and size. In all cases, the content of the

coating was 0.97-0.98 wt.%, which was achieved by carefully preparing the sol-gel coating with a high reproducibility; the deviation for the 10 coating experiments from the mean coating content was 0.49-1% for all the studied powders with different particle size and shape. The phase composition of the coating was analyzed by X-ray analysis. The apparent and tap densities were estimated (ISO 4490, ISO 3953) and the ratio tap density/apparent density was calculated (friction index).

Cylindrical specimens $\varnothing 10 \times 5$ mm³ were cold pressed in a hard-metal die at uni-axial pressures from 50 to 800 MPa (3-5 specimens at each pressure); the density/porosity was determined by weighing and measuring the dimensions of the specimens with the accuracy of ± 0.0001 g and ± 0.005 mm. The compressibility of the powders was evaluated by the compression ratio ($CR = \rho_{app}/\rho_{teor}$), by relative density ($RD = \rho/\rho_{teor}$) and by porosity P (%) achieved at the pressing pressure "p". The compressibility was quantified using the compaction equation formulated by Parilák et al. [14]:

$$P = P_o \cdot \exp(-K \cdot p^n) \quad (1)$$

P_o (%) is the apparent porosity calculated from apparent density. Parameter K is related to powder particle morphology and parameter n is related to the ability of powder particles for densification by plastic deformation. The equation (1) enables one to calculate compressibility parameters that characterize the behaviour of metal powders during the compaction, as are:

Parameter "X_n" (for $P \rightarrow 0$ at $p \rightarrow \infty$):

$$X_n = \frac{P_o}{n} \left[\sqrt{2 \cdot \pi \cdot n} / (e \cdot \pi \cdot K)^{1/n} \right] \cdot \left[1 - \omega \left(\frac{1}{n} \right) \right], \text{ where } \omega \left(\frac{1}{n} \right) \leq e^{n/2} - 1 \quad (2)$$

which expresses the value of total „work“ required for the densification of the compact to full density through a rearrangement (shift and rotation) and plastic deformation of the particles. Its value corresponds to the area under the compaction curve, until to $P = 0$.

Parameter "X₁" (for $n = 1$): $X_1 = P_o/K$

(3)

is that part of the "work" that is necessary for the densification of the compact through a rearrangement of particles (by shift and rotation) without plastic deformation development (for $n = 1$). It is a "fictitious" value, which corresponds to the area under the compaction curve defined by a fictitious porosity P_1 . This assumption is an approximation, since the relocation of particles that have the ability to deform plastically, is always associated with a minimal plastic deformation of the surface roughness.

Pressure "p₁":

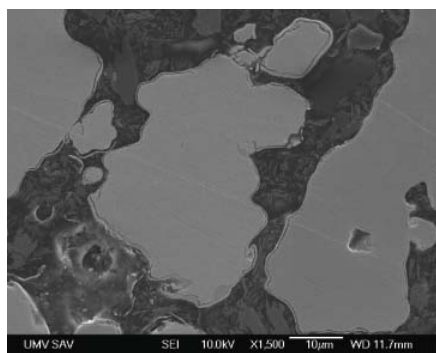
$$p_1 = [\ln(P_o) - \ln(P)] \cdot 1/K \quad (4)$$

which is a "fictitious" value of pressure (fictitious because it can only be obtained by calculation; a real one is not measurable) and reflects the pressure „p“ needed for densification up to $P_1 = 0$. The value of the p_1 expresses the pressure at which is finished the densification dominantly by rearrangement (shift and rotation) and starts to dominate the densification by development of plastic deformation. At the pressure p_1 is reached a total porosity P_{p1} , which may be related to the state of geometric hardening of the powder system. Further densification with increasing pressure will take place by plastic deformation.

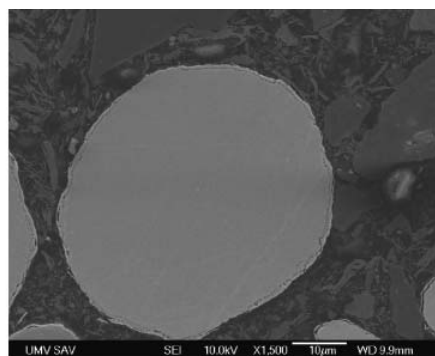
The microstructure of the compacts was studied by optical microscopy.

RESULTS AND DISCUSSION

Figures 2a,b show the metallographic section of the Fe/SiO₂ particles of the NPB and MPB powders. The coating is continuous with a thickness of 3000-5000 nm and has good cohesion with the surface of Fe particles. Defects were only sporadically identified; those such as cracking or inconsistencies, occurred especially in the case of irregularly shaped Fe particles. It should be noted that the coating on the surface of the Fe particle is also composed of Fe oxide phases that are modified by the heat treatment used. After calcination at 400°C/air the layer consists of the phases: amorphous SiO₂, α -Fe₂O₃, Fe₃O₄ which have been predicted by ThermoCalc calculation and identified by X ray analysis, Fig.3, Fig.4. The ratio of the Fe-oxide was determined by chemical analysis of the Fe²⁺ and Fe³⁺ contents and up to ~ 30 wt.%.



a)



b)

Fig.2. Metallographic section of the Fe/SiO₂ coated particle a) with irregular shaped Fe particles (NPB), b) with spherical shaped Fe particles (MPB).

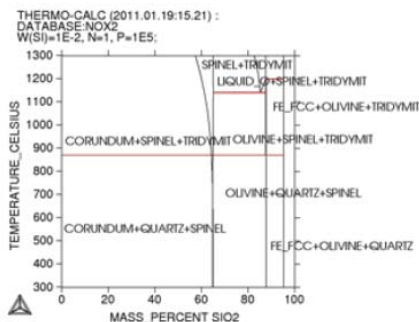


Fig.3. Theoretical phase diagram Fe₂O₃-SiO₂, ThermoCalc Classic 4.0.

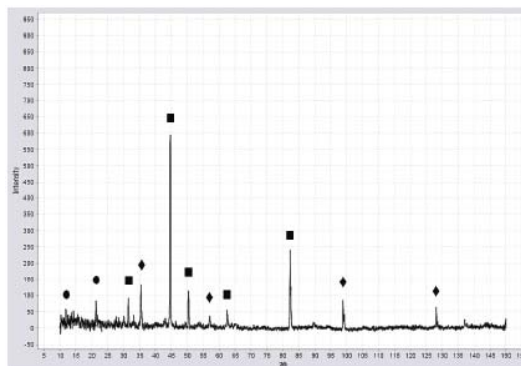


Fig.4. X-ray analysis of the coating calcined at 400°C (♦Fe₃O₄ ●SiO₂ ■ Fe₂O₃).

Table 3 summarizes the values of the theoretical density, apparent and tap densities, friction index and apparent porosity of the Fe/SiO₂ tested powders. It is visible that the apparent density of powders with irregular shaped Fe particles increases, while in the case of spherical particles there is the tendency for a slight decrease. A similar trend is also in the case of tap density. Silane addition leads to a slight increase of apparent density but slightly reduces the tap density which results in a slight decrease in friction index

values. Data summarized in Tables 4-6 show the relative density of the compacts in dependence on the compacting pressure. It is seen that the shape of Fe particles strongly influences the development of the relative density of compacts. As a result of higher apparent density the relative density is higher for the powders with spherical particle shape (48-55% when compared with 36-39% for irregular shape), particularly in the area of lower pressing pressures. The values of the relative density at a pressure of 800 MPa are in the range of 84-88%. The addition of Silane has a significant positive impact on the relative density, which, after pressing pressure 800 MPa reaches 93.5-96.5%. Figures 5a,b,c show the influence of both the shape and size of Fe particles and Silane addition to Fe/SiO₂ powders on the relative density of the compacts based on a) NP and MP powders, b) N1 and M1 powders, c) N2 and M2 powders. It can be seen that the joint effect of coating as well as the addition Silane was shown for powder based monodispersed Fe powders.

Tab.3. The basic physical properties of the Fe/SiO₂ powders.

| Powder | ρ_{theor} [g/cm ³] | ρ_{app} [g/cm ³] | ρ_{tap} [g/cm ³] | Friction index | P_0 [%] |
|--------|---|---|---|----------------|--------------|
| NP | 7.845 | 2.965 | 3.733 | 1.252 | 62.205 |
| NPB | 7.783 | 3.107 | 3.982 | 1.281 | 60.080 |
| NPB+S | 7.245 | 2.999 | 3.572 | 1.193 | 58.577 |
| N1 | 7.845 | 2.798 | 3.576 | 1.279 | 64.334 |
| N1B | 7.783 | 2.989 | 3.602 | 1.205 | 61.596 |
| N1B+S | 7.245 | 2.897 | 3.677 | 1.267 | 59.986 |
| N2 | 7.845 | 2.811 | 3.677 | 1.306 | 64.145 |
| N2B | 7.783 | 2.823 | 3.553 | 1.258 | 63.729 |
| N2B+S | 7.243 | 2.925 | 3.902 | 1.339 | 59.669 |
| MP | 7.845 | 4.623 | 5.032 | 1.093 | 41.315 |
| MPB | 7.789 | 4.132 | 5.099 | 1.234 | 46.916 |
| MPB+S | 7.246 | 3.988 | 4.501 | 1.130 | 45.028 |
| M1 | 7.845 | 4.147 | 4.840 | 1.169 | 47.138 |
| M1B | 7.783 | 4.108 | 4.762 | 1.159 | 47.218 |
| M1B+S | 7.245 | 4.333 | 5.072 | 1.174 | 40.193 |
| M2 | 7.845 | 3.752 | 5.560 | 1.154 | 52.173 |
| M2B | 7.783 | 3.774 | 4.589 | 1.215 | 51.512 |
| M2B+S | 7.243 | 3.874 | 4.868 | 1.255 | 46.547 |

Tab.4. Relative density of compacts from NP, NPB, NPB+S, MP, MPB, MPB+S powders.

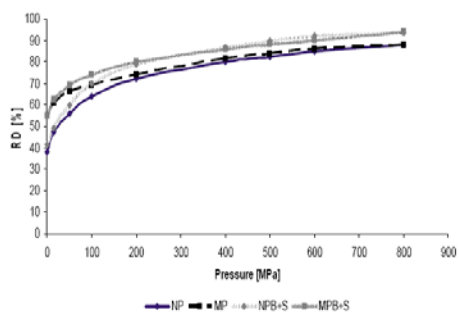
| Pressure [MPa] | Relative density [%] | | | | | |
|----------------|----------------------|------|-------|------|------|-------|
| | NP | NPB | NPB+S | MP | MPB | MPB+S |
| 0 | 37.8 | 39.9 | 41.4 | 55.0 | 53.1 | 55.0 |
| 15 | 47.5 | 51.2 | 49.3 | 61.0 | 61.0 | 62.3 |
| 50 | 56.0 | 59.0 | 59.8 | 66.0 | 66.2 | 69.0 |
| 100 | 64.0 | 66.0 | 70.0 | 69.0 | 70.6 | 74.0 |
| 200 | 72.0 | 72.0 | 79.0 | 74.0 | 76.0 | 80.0 |
| 400 | 80.1 | 80.0 | 87.0 | 81.5 | 81.0 | 86.1 |
| 500 | 82.5 | 82.0 | 90.0 | 84.0 | 82.5 | 88.4 |
| 600 | 85.0 | 84.5 | 92.0 | 86.0 | 84.0 | 90.4 |
| 800 | 88.0 | 85.4 | 93.5 | 88.1 | 86.0 | 93.9 |

Tab.5. Relative density of compacts based on N1, N1B, N1B+S, M1, M1B, M1B+S powders.

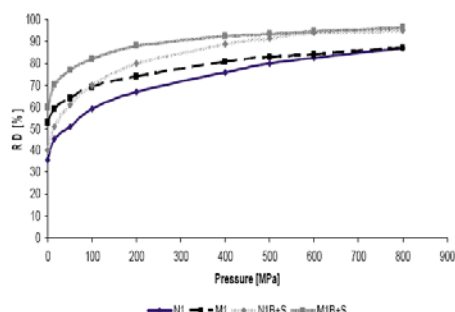
| Pressure [MPa] | Relative density. % | | | | | |
|----------------|---------------------|------|-------|------|------|-------|
| | N1 | N1B | N1B+S | M1 | M1B | M1B+S |
| 0 | 35.7 | 38.4 | 40.0 | 52.9 | 52.8 | 59.8 |
| 15 | 45.5 | 46.3 | 50.8 | 59.1 | 61.2 | 70.1 |
| 50 | 51.0 | 52.3 | 60.8 | 64.0 | 66.3 | 77.0 |
| 100 | 59.0 | 59.0 | 70.0 | 69.0 | 70.0 | 82.0 |
| 200 | 67.0 | 67.0 | 80.0 | 74.0 | 74.0 | 88.0 |
| 400 | 76.0 | 77.0 | 89.0 | 80.6 | 78.8 | 92.4 |
| 500 | 80.0 | 80.0 | 91.5 | 82.8 | 81.0 | 93.8 |
| 600 | 82.6 | 82.0 | 94.0 | 84.0 | 82.3 | 95.0 |
| 800 | 87.0 | 85.0 | 95.2 | 87.1 | 86.0 | 96.5 |

Tab.6. Relative density of compacts based on N2, N2B, N2B+S, M2, M2B, M2B+S powders.

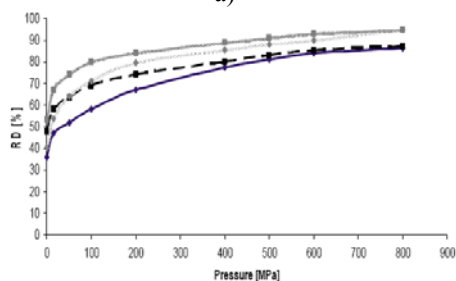
| Pressure [MPa] | Relative density. % | | | | | |
|----------------|---------------------|------|-------|------|------|-------|
| | N2 | N2B | N2B+S | M2 | M2B | M2B+S |
| 0 | 35.9 | 36.3 | 40.3 | 47.9 | 48.5 | 53.5 |
| 15 | 47.0 | 45.7 | 53.9 | 58.2 | 57.0 | 67.2 |
| 50 | 52.0 | 52.0 | 64.0 | 63.4 | 63.0 | 74.0 |
| 100 | 58.0 | 57.0 | 71.0 | 69.0 | 68.0 | 80.0 |
| 200 | 67.0 | 65.0 | 79.6 | 74.0 | 75.0 | 84.3 |
| 400 | 77.3 | 75.0 | 85.6 | 79.8 | 81.0 | 89.0 |
| 500 | 81.0 | 78.0 | 88.1 | 83.0 | 83.0 | 91.0 |
| 600 | 84.0 | 80.0 | 89.9 | 85.0 | 86.0 | 93.0 |
| 800 | 86.4 | 84.0 | 95.0 | 87.0 | 89.7 | 95.0 |



a)



b)



c)

Fig.5. The influence of both shape and size of Fe particles and Silane addition coated powder on relative density of the compacts based on (a) NP and MP powders, (b) N1 and M1 powders, (c) N2 and M2 powders.

In Tables 7-9 are the values of compression ratio for the tested powders. Lower values of the compression ratio of powders with spherical particles correspond to a higher apparent density. Silane addition slightly increases the compression ratio in the full compression pressure range. This effect is more pronounced in the case of a monodisperse of powders.

Tab.7. Compression ratio of the NP, NPB, NPB+S and MP, MPB, MPB+S powders.

| Pressure [MPa] | Compression ratio | | | | | |
|----------------|-------------------|-----|-------|-----|-----|-------|
| | NP | NPB | NPB+S | MP | MPB | MPB+S |
| 0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 |
| 15 | 1.3 | 1.3 | 1.2 | 1.2 | 1.1 | 1.1 |
| 50 | 1.5 | 1.4 | 1.4 | 1.3 | 1.2 | 1.3 |
| 100 | 1.7 | 1.7 | 1.7 | 1.3 | 1.3 | 1.3 |
| 200 | 1.9 | 1.8 | 1.9 | 1.5 | 1.4 | 1.5 |
| 400 | 2.1 | 2.0 | 2.1 | 1.5 | 1.5 | 1.6 |
| 500 | 2.2 | 2.1 | 2.2 | 1.6 | 1.6 | 1.6 |
| 600 | 2.2 | 2.1 | 2.2 | 1.6 | 1.6 | 1.6 |
| 800 | 2.3 | 2.1 | 2.3 | 1.7 | 1.6 | 1.7 |

Tab.8. Compression ratio of the N1, N1B, N1B+S and M1, M1B, M1B+S powders.

| Pressure [MPa] | Relative density [%] | | | | | |
|----------------|----------------------|-----|-------|-----|-----|-------|
| | N1 | N1B | N1B+S | M1 | M1B | M1B+S |
| 0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 |
| 15 | 1.3 | 1.2 | 1.3 | 1.1 | 1.2 | 1.2 |
| 50 | 1.4 | 1.4 | 1.5 | 1.2 | 1.3 | 1.3 |
| 100 | 1.7 | 1.5 | 1.7 | 1.3 | 1.3 | 1.4 |
| 200 | 1.9 | 1.7 | 2.0 | 1.4 | 1.4 | 1.5 |
| 400 | 2.1 | 2.0 | 2.2 | 1.5 | 1.5 | 1.5 |
| 500 | 2.2 | 2.1 | 2.3 | 1.6 | 1.5 | 1.6 |
| 600 | 2.3 | 2.1 | 2.3 | 1.6 | 1.6 | 1.6 |
| 800 | 2.4 | 2.2 | 2.4 | 1.6 | 1.6 | 1.6 |

Tab.9. Compression ratio of the N2, N2B, N2B+S and M2, M2B, M2B+S powders.

| Pressure [MPa] | Compression ratio | | | | | |
|----------------|-------------------|-----|-------|-----|-----|-------|
| | N2 | N2B | N2B+S | M2 | M2B | M2B+S |
| 0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 |
| 15 | 1.3 | 1.3 | 1.3 | 1.2 | 1.2 | 1.3 |
| 50 | 1.5 | 1.4 | 1.6 | 1.3 | 1.3 | 1.4 |
| 100 | 1.6 | 1.6 | 1.8 | 1.4 | 1.4 | 1.5 |
| 200 | 1.9 | 1.8 | 2.0 | 1.5 | 1.5 | 1.6 |
| 400 | 2.2 | 2.1 | 2.1 | 1.7 | 1.7 | 1.7 |
| 500 | 2.3 | 2.2 | 2.2 | 1.7 | 1.7 | 1.7 |
| 600 | 2.3 | 2.2 | 2.2 | 1.8 | 1.8 | 1.7 |
| 800 | 2.4 | 2.3 | 2.4 | 1.8 | 1.9 | 1.8 |

Figures 6a-c display the compaction curves for the coated powders with irregular and spherical particles. Figures 7a-c show the compaction curves for the same powders

with the addition of a Silane binder. The traces in the diagrams are the representation of the compaction equations (1) and (4) which quantify the compressibility of the powders; the correlation coefficients obtained by fitting the experimental data with equation (1) are in the range of 0.967-0.9982 which means that the equation (1) can be also considered as applicable for the coated powders studied. The values of the compaction parameters p_1 , P_{p1} , X_n , X_1 , P_{800} , whose meaning was given in the experimental section, are in Table 10.

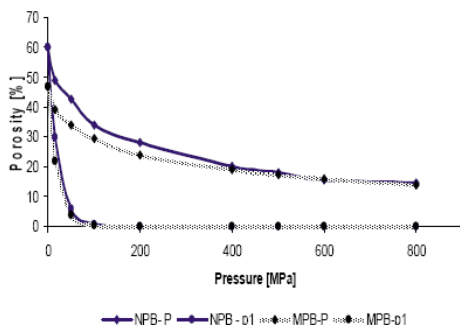


Fig.6a. Compaction curves for NPB, MPB powders:

NPB $60.09 \exp(-0.047 \cdot p^{0.5284})$; $r = 0.9704$
 MPB $46.91 \exp(-0.051 \cdot p^{0.5057})$; $r = 0.9982$
 NP $62.21 \exp(-0.0367 \cdot p^{0.5818})$; $r = 0.9859$

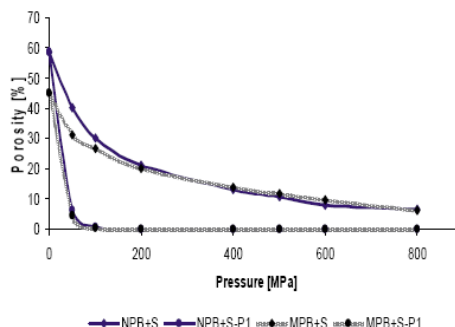


Fig.7a. Compaction curves for NPB+S, MPB+S powders:

NPB+S $58.58 \exp(-0.035 \cdot p^{0.6509})$; $r = 0.9567$
 MPB+S $45.03 \exp(-0.034 \cdot p^{0.6047})$; $r = 0.9849$
 MP $43.13 \exp(-0.0283 \cdot p^{0.5889})$; $r = 0.9738$

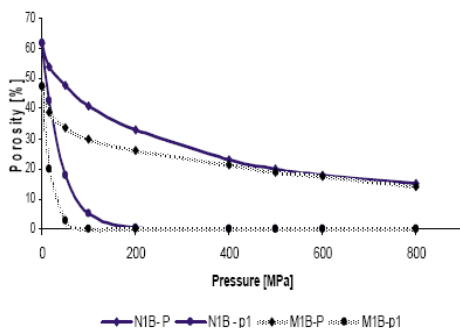


Fig.6b. Compaction curves for N1B, M1B powders:

N1B $61.569 \exp(-0.026 \cdot p^{0.6195})$; $r = 0.9759$
 M1B $47.218 \exp(-0.057 \cdot p^{0.5025})$; $r = 0.9884$
 N1 $64.334 \exp(-0.032 \cdot p^{0.5796})$; $r = 0.9888$

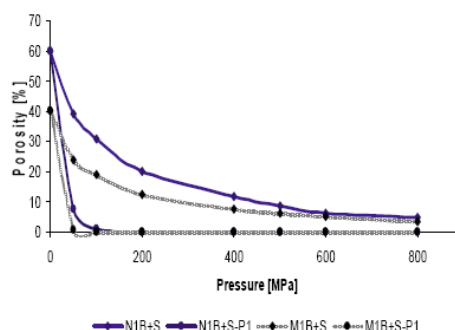


Fig.7b. Compaction curves for N1B+S, M1B+S powders:

N1B+S $59.986 \exp(-0.0324 \cdot p^{0.6693})$; $r = 0.9665$
 M1B+S $40.193 \exp(-0.0679 \cdot p^{0.5435})$; $r = 0.9775$
 M1 $45.030 \exp(-0.0296 \cdot p^{0.5743})$; $r = 0.9651$

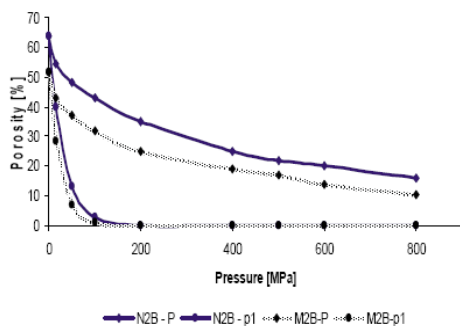


Fig.6c. Compaction curves for N2B, M2B powders:

$$\begin{aligned} \text{N2B } 63.729 \exp(-0.0314 \cdot p^{0.5726}); r = 0.9683 \\ \text{M2B } 51.519 \exp(-0.0386 \cdot p^{0.5680}); r = 0.9932 \\ \text{N2 } 64.145 \exp(-0.0182 \cdot p^{0.6830}); r = 0.9624 \end{aligned}$$

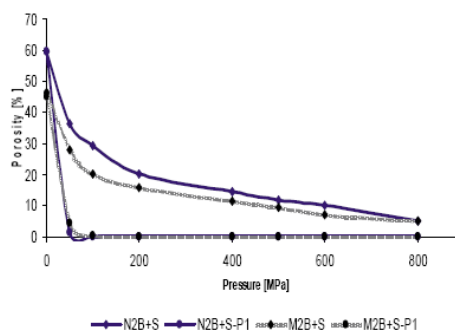


Fig.7c. Compaction curves for N2B+S, M2B+S powders:

$$\begin{aligned} \text{N2B+S } 59.669 \exp(-0.0576 \cdot p^{0.5532}); r = 0.9781 \\ \text{M2B+S } 46.547 \exp(-0.0898 \cdot p^{0.5011}); r = 0.9902 \\ \text{M1 } 52.147 \exp(-0.0501 \cdot p^{0.5050}); r = 0.9789 \end{aligned}$$

Tab.10. Values of the compaction parameters p_1 , P_{p1} , X_n , X_1 , P_{800} .

| Powder | Type | P_0 [%] | p_1 [MPa] | P_{p1} [%] | X_1 | X_n | P_{800} [%]* |
|--------|-----------------------------------|-----------|-------------|--------------|-------|-------|----------------|
| NP | uncoated | 62.205 | 238 | 25.6 | 17 | 296 | 12.0 |
| MP | | 43.133 | 297 | 20.6 | 16 | 303 | 10.1 |
| NPB | coated | 60.088 | 186 | 28.9 | 13 | 374 | 14.6 |
| MPB | | 46.912 | 165 | 24.3 | 10 | 372 | 13.4 |
| N1 | uncoated | 64.334 | 287 | 28.3 | 20 | 374 | 12.9 |
| M1 | | 45.030 | 285 | 22.0 | 16 | 330 | 11.8 |
| N1B | coated | 61.569 | 355 | 24.2 | 25 | 328 | 17.0 |
| M1B | | 47.218 | 148 | 23.6 | 8 | 281 | 14.7 |
| N2 | uncoated | 64.145 | 481 | 18.6 | 35 | 258 | 13.6 |
| M2 | | 52.147 | 171 | 26.6 | 10 | 371 | 12.6 |
| N2B | coated | 63.729 | 279 | 29.0 | 20 | 410 | 15.9 |
| M2B | | 51.519 | 222 | 23.6 | 13 | 284 | 10.2 |
| NPB+S | coated + 1.0 wt.% Silane | 58.577 | 266 | 17.1 | 18 | 137 | 6.4 |
| MPB+S | | 45.028 | 242 | 17.0 | 13 | 161 | 6.0 |
| N1B+S | | 59.986 | 268 | 15.2 | 18 | 114 | 4.0 |
| M1B+S | | 40.193 | 122 | 15.9 | 6 | 92 | 5.0 |
| N2B+S | | 59.669 | 150 | 23.0 | 10 | 164 | 5.0 |
| M2B+S | | 46.547 | 94 | 19.0 | 5 | 108 | 5.0 |

* P_{800} means the porosity achieved at the pressing pressure of 800 MPa.

There is a clear difference between the compaction curves for powders NPB and MPB. Figure 6a with irregular and spherical particles at low pressures which corresponds to the densification by rearrangement of particles (shift and rotation). It is obvious that a denser arrangement of spherical particles already in the „free-pouring” state heavily restricts the processes of particle rearrangement. However, the friction between the particles is also acting; the surface roughness causes that, while the values of the pressure p_1 (Tab.10) are about the same, the values of porosity P_{p1} at p_1 and of the work (X_1) needed for

densification by rearrangement of particles are lower for the spherical particles. The value of the „work“ X_n needed for densification by plastic deformation is comparable for both powders. Comparing the values of X_n with those of the same uncoated powders (NP, MP, Tab.10) shows that the values are lower for uncoated powders. This reflects the negative influence of coating on powder densification by plastic deformation which results in the slightly higher porosity reached at $p = 800$ MPa for the coated powders, while the particle shape does not have significant effect on compressibility behaviour.

The addition of Silane to both powders (NPB+S and MPB+S), Fig.7a, increases the value of p_1 but decreases the value of P_{p1} which means that Silane has a positive effect on the densification in the stage of rearrangement of particles by shift and rotation. A more dense arrangement of the particles enables a more effective development of plastic deformation resulting in a significant decrease of porosity, up to 6-6.4% for $p = 800$ MPa (Tab.10).

The difference between the compaction behaviour of irregular and spherical particles is also observed in the case of monodisperse powders N1B and M1B (Fig.6b). Thus the pressure p_1 and the values of P_{p1} are significantly higher for powders with irregular particles than with spherical particles (Tab.10). Data in Tab.10 show that the parameter X_1 is much smaller for powders with spherical particles than for those with irregular particles. For pressures higher than p_1 the compaction curves are similar for both powders - the densification is controlled by the ability of the iron core to deform plastically. The porosity achieved for $p = 800$ MPa is lower for the powder with spherical particles. As for the polydisperse powders (NPB, MPB), the addition of a Silane binder, Fig.7b, has a positive effect on particle rearrangement (lower value of X_1 , Tab.10). Following a denser arrangement of particles, the plastic deformation of iron particles is more effective and the densification is greater which results in lower porosity of 4.8-5.0% for $p = 800$ MPa.

The monodisperse coated powders with fine particles (N2B, M2B), Fig.6c, showed a compaction behaviour comparable to the powders N1B and M1B. Comparing the M1B and M2B powders (Tab.10) with spherical particles of a different size, the pressure p_1 is shifted to a higher value for finer particles, practically at the same value of P_{p1} but at higher X_1 value; the resulting porosity for $p = 800$ MPa is lower for the powder M2B with fine spherical particles (10.2%) than for M1B (14.7%). The addition of Silane (Fig.7c) reduces the value X_1 , mainly for spherical particles. Due to a denser arrangement of particles and thus a more effective development of plastic deformation (lower X_n values), the addition of Silane leads to a lower porosity of 5% for $p=800$ MPa, for both powders with irregular and spherical particles when compared with powders without the Silane addition.

Figures 8a,b and 9a,b show examples of the microstructure of compacts based on the powders N1B+S and M1B+S pressed at the pressures of 400 and 800 MPa. It is seen that the coating forms a continuous network without microscopically obvious cracks or other damage even when using a high compacting pressure. The continuous network in the compact shows a good bond of the coating with the iron particle, even when handling the powder and during compaction as well. The morphology of the network is determined by the shape of the deformed iron powder particles.

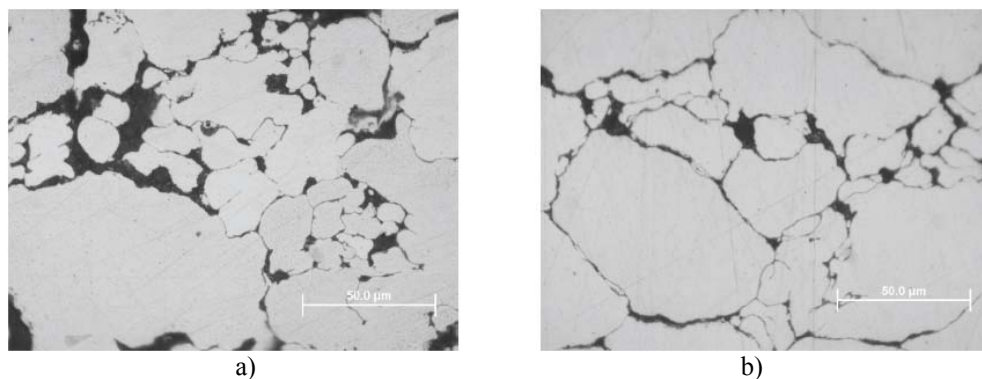


Fig.8. The microstructure of the compact based on the Fe/SiO₂ powder with irregular shaped Fe particles and with addition of Silane (N1B+S), compacted at (a) 400 MPa, (b) 800 MPa.

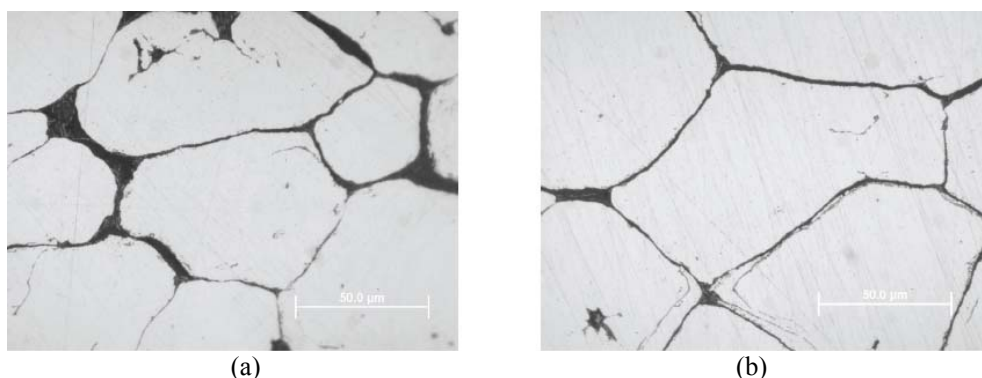


Fig.9. The microstructure of the compact based on the Fe/SiO₂ powder with spherical Fe particles and with addition of Silane (M1B+S), compacted at (a) 400 MPa, (b) 800 MPa.

The microstructure of the compacts resulting from the pressure of 400 MPa is comprised of residual pores; also some local plastic deformation can be identified, particularly in areas of particle contacts. The compaction at the pressure of 800 MPa, which corresponds to the relative density of 95% for both presented powders results in a disappearance of large pores that are filled with surface roughness and fine particles. The densification process is supported by the positive effect of Silane, as well as by localized plastic deformation and intensive volume plastic deformation of Fe particles. The insulating coating follows well the surface of deformed particles.

CONCLUSIONS

The coating has a slightly negative effect on the compressibility, which is reflected in the slightly higher values of the porosity after compaction at a pressing pressure of 800 MPa when compared with the same uncoated powders.

After exhausting the possibilities of rearrangement of particles by shift and rotation (geometrical hardening state), the densification is further controlled by plastic deformation of the iron core inside the composite particles.

The differences between the compressibility of powders with irregular and spherical particles were identified in the area of low pressures which corresponds to the densification through particle rearrangement (shift and rotation). A denser arrangement of spherical particles, already in the loose state, heavily restricted the relocation processes.

The addition of 1.0 wt.% Silane binder to the coated powders with irregular and spherical particles has a significantly positive effect on compressibility. The decrease of porosity after compaction at the pressing pressure 800 MPa is two to three-folds when compared to the same coated powders without the binder addition.

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