EFFECT OF COMPACTING PRESSURE ON INTERCONNECTED POROSITY IN IRON PM COMPACTS

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Abstract
The purpose of this work was to analyse the influence of compacting pressure on interconnected porosity in iron compacts, both in macro- and microscale. Size and volume fraction of micro- meso- and macropores were examined in compacts pressed under pressure within 200 to 400 MPa, made in laboratory conditions of iron powder ASC 100.29 manufactured by Höganäs AB company. The interconnected porosity was determined using a method based on measuring the sorption isotherms of CO_2 and benzene at T = 25°C in static conditions in a high-vacuum gravimetric appliance equipped with McBain-Bakr weighers.
Relationships between compacting pressure and the interconnected micro- and macroporosity of the examined compacts were determined.
Keywords: interconnected micro- and macroporosity, compacting pressure, iron compacts

INTRODUCTION

The interconnected porosity is a part of the total porosity of sintered products. The interconnected pores, occurring in ferrous compacts with density below 7.3 g.cm^{-3} besides the closed pores, form a labyrinth of tiny, interconnected capillary conduits. They play a particular role in porous self-lubricating bearings, filters etc. Of equal importance is their effect in the products subject to thermochemical treatment processes, where they create specific paths for gas penetration into the material.

As in the case of total porosity, the degree of interconnected porosity, distribution of pores in the material and their size depend on numerous factors, of which the main ones are compaction pressure, temperature and time of sintering, quality of metal powder and manner of its manufacture, particle size and distribution, as well as all the factors affecting pressure distribution in the compact [1-4]. Although, with respect to the application, the most important and conclusive issue is porosity of a finished product after sintering, it is necessary to evaluate and analyse it as early as at the stage of powder manufacture and compacting, to allow obtaining a controlled porosity. So far, no mathematical relationship between interconnected porosity and size, shape and sieve analysis of powder has been developed. General empirical relationships are known, concerning proper selection of metal powders to obtain the required final properties of a designed product. It is also commonly known that the most important factor controlling the porosity degree, including the interconnected porosity, is the compacting pressure.

In this work, the effect of compacting pressure on interconnected porosity in iron compacts, prepared in laboratory conditions of iron powder ASC 100.29 (manufactured by Swedish company Höganäs AB), was investigated. The interconnected porosity was determined in macro- and microscopic scale.

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EXPERIMENTAL MATERIALS AND METHODS

The compacts for examination, a cylinder diameter 13 mm x length 8 mm and a ring OD 10 mm x ID 5 mm x thickness 4 mm, were prepared of iron powder ASC 100.29 (manufactured by Höganäs AB). The sieve analysis and shape of particles of selected iron powder are shown in Table 1 and Fig.1, respectively.

Tab.1. Sieve analysis of selected iron powder [5].

Iron Powder	Sieve analysis [%]			
	< 45 μm	45 ÷ 150 μm	150 ÷ 180 μm	> 180 μm
ASC 100.29	23	69	8	0

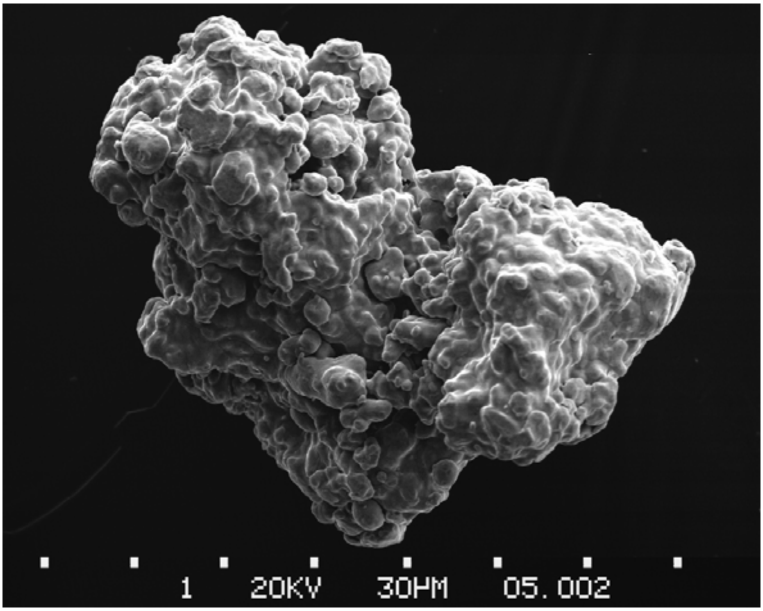

Fig.1. An irregular, somewhat rounded particle of iron powder ASC 100.29. SEM.

The test specimens were one-side compacted and the applied pressure was within 200 to 400 MPa. For each compacting pressure, three compacts were prepared, in the form of cylinders and rings.

The interconnected porosity was determined in macro- and microscopic scale. The method used for determining the macroscopic porosity consisted of measuring the mass increase after impregnation with oil, according to EN ISO 2738:1999.

The interconnected porosity in microscopic scale was determined by measurements of the sorption isotherms of CO_2 and benzene at T = 25°C in static conditions, in a high-vacuum gravimetric appliance equipped with McBain-Bakr weighers (home-designed appliance). The Dubinin-Raduschkevitch theory of volumetric micropores filling was applied for determining the pores volume from CO_2 sorption, and the capillary condensation theory with the Kelvin equation was applied for interpreting the benzene sorption isotherms.

The specimens for interconnected porosity examination were taken from the ring-shaped compacts OD 10 mm x ID 5 mm x thickness 4 mm. The rings were broken into 4 approximately equal pieces, each of ca. 0.5 g, and one of the pieces was selected for the measurement.

The measurements were carried out on three above-described specimens taken from three ring-shaped compacts pressed under different pressures of 200, 300 and 400 MPa.

RESULTS AND DISCUSSION

The interconnected macroporosity of the compacts, determined by the method of mass increase after impregnation with oil (acc. to EN ISO 2738:1999), is given in Table 2.

Tab.2.Test results of interconnected porosity (macropores) in iron compacts.

Test specimen	Compacting pressure [MPa]	„Green" density [$g.cm^{-3}$]	Interconnected porosity* [%]	Interconnected porosity* [$cm^3.g^{-1}$]
ASC-200	200	5.6	24.5	0.0436
ASC-300	300	6.0	20	0.0333
ASC-400	400	6.4	15.5	0.0243
*average results of 3 measurements				

The interconnected microporosity (micro- and mesopores) of the compacts, determined by measurements of CO_2 and benzene sorption isotherms, is given in Table 3.

Tab.3. Test results of interconnected porosity (micro- and mesopores) in iron compacts.

Test specimen	Compacting pressure [MPa]	Green density [$g.cm^{-3}$]	Micropores		Mesopores				Micro- and mesopores
			Volume V [$cm^3.g^{-1}$]		Volume V [$cm^3.g^{-1}$]				Volume V [$cm^3.g^{-1}$]
			< 0.4 nm	0.4-2 nm	2-3 nm	3-5 nm	5-10 nm	10-50 nm	< 50 nm
ASC-200	200	5.6	0.0002	0.0000	0.0000	0.0002	0.0009	0.0054	0.0067
ASC-300	300	6.0	0.0000	0.0001	0.0005	0.0006	0.0025	0.0070	0.0107
ASC-400	400	6.4	0.0012	0.0004	0.0006	0.0005	0.0014	0.0081	0.0122

Volume distribution of micro- and mesopores in the iron compacts, for the compacting pressures of 200, 300 and 400 MPa is shown in Fig.2.

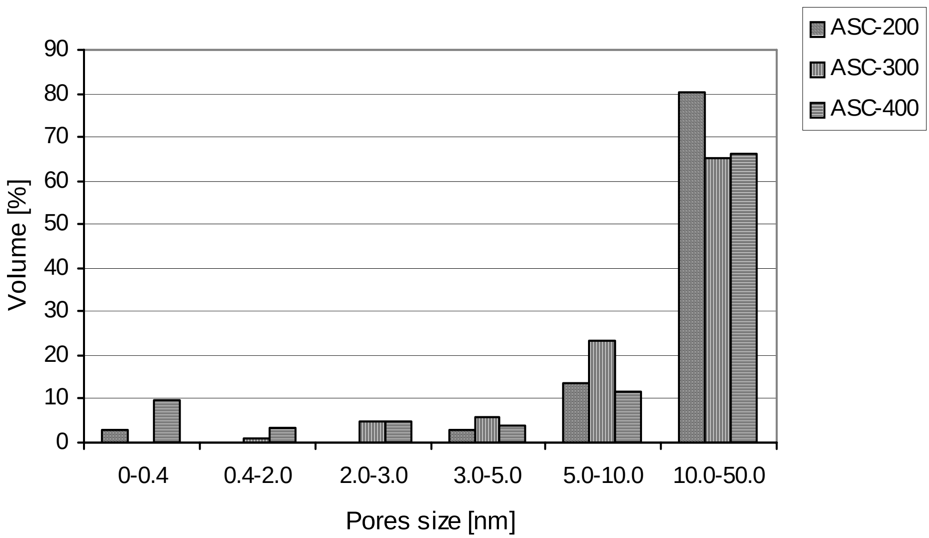

Fig.2. Volume distribution of interconnected micro- and mesopores in compacts of iron powder ASC 100.29 for compacting pressure 200, 300 and 400 MPa.

Analysis of size and volume distribution of micro- and mesopores in the iron compacts reveals the following:

- The largest volume portion of micropores (0.4 to 2 nm) occurs in the compacts pressed under the highest pressure of 400 MPa. There are practically no micropores in the compacts pressed under pressures 200 and 300 MPa.
- In all the compacts (200, 300 and 400 MPa), portion of the mesopores increases with their increasing size, in a relatively regular way. Most of the mesopores are within the diameter range of 10 to 50 nm.

The influence of applied compacting pressure on interconnected macro- and micro-porosity (micro- and mesopores together) is shown in Figs.3 and 4, respectively. The measured points plotted on the graphs are approximated by a polynomial of degree two.

It is characteristic, that with an increasing of compacting pressure, within 200 to 400 MPa, the volume of interconnected macropores decreases and the volume of micro- plus mesopores increases. The run of approximated curve on the diagram, illustrated the effect of compacting pressure on interconnected microporosity (Fig.4), indicates that after reaching the pressure of 400 MPa the volume of micro- plus mesopores drops. It seems that higher compacting pressure also causes a change in volume distribution of interconnected microporosity. Probably, with an increasing of compacting pressure, the volume fraction of micropores (with size lower than 2 nm) also increases, in turn, volume fraction of mesopores decreases. This trend is already visible in examined iron compacts, pressed under pressure from 200 to 400 MPa (see Fig.2). These assumptions still require experimental verification.

In light of the obtained results, it is worth mentioning the changes in relative volume fraction of interconnected pores (micro-, meso- and macropores) which will occur during sintering the examined compacts. As a result of the structure non-homogeneity demonstrated by local fluctuations of density and porosity, the densification process during sintering will proceed in a differentiated way. According to [6], at initial sintering stages a growth of large pores i.e. macropores (>50 nm) is observed, accompanied by reduced fraction of mesopores (<50 nm) and decay of micropores (<2 nm). In the subsequent intermediate sintering stages, all the interconnected pores will be fairly equally eliminated. The final size and number of interconnected pores, at a constant temperature directly dependent on the sintering time, will be conditioned by the desired sintered compact density.

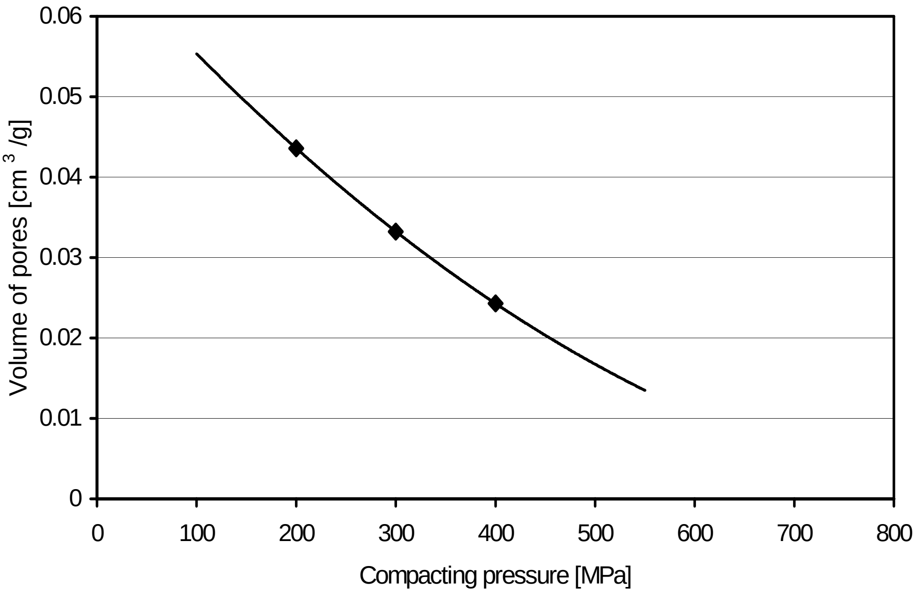

Fig.3. Interconnected macroporosity vs. compacting pressure in iron compacts.

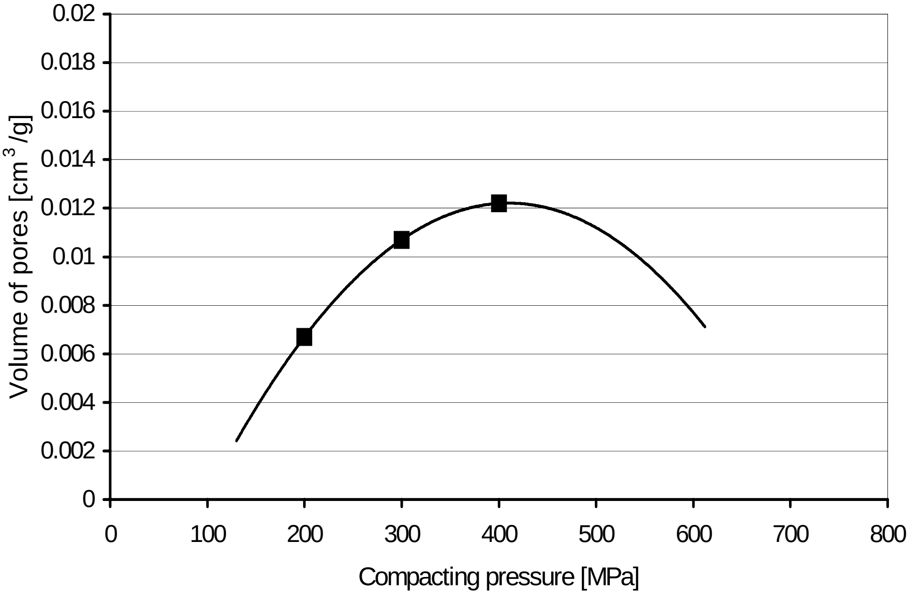

Fig.4. Interconnected microporosity vs. compacting pressure in iron compacts.

CONCLUSIONS

In the microscopic scale, the growth of compacting pressure, in the examined range, results in the increase of interconnected porosity. The quantitative analysis of interconnected microporosity carried out on all iron compacts proved that the greatest volume of micro- plus mesopores (micro- and mesopores together) occurs in iron compacts pressed under the highest applied pressure.

In the macroscopic scale, the increase of compacting pressure in the examined range causes reduction of interconnected porosity. The volume fraction of interconnected macropores decreases in iron compacts with an increasing of compacting pressure.

References

[1] German, RM.: Powder Metallurgy Science. 2nd ed. Princeton : Metal Powder Industries Federation, 1994
[2] F. Lenel: Powder Metallurgy, Principles and Applications. Princeton : Metal Powder Industries Federation, 1980
[3] Powder Metal Technologies and Applications. Vol. 7. ASM Handbook, 10 ed. ASM International, 1998
[4] Höganäs Handbook for sintered components. Handbook 2. Höganäs AB, 1997
[5] Höganäs Handbook for Iron and Steel Powders. Höganäs AB., 2002
[6] Lis, J., Pampuch, R.: Prace Kom. Ceram. O/PAN, Kraków, vol. 36, 1988, p. 7