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COMPACTION AND PROPERTIES OF HIGHLY POROUS POWDER PARTS PRODUCED WITH VARIOUS PORE FORMERS

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ABSTRACT

The powder metallurgy route for manufacturing highly porous iron based materials was considered. To increase the porosity, the powders of ammonium, bicarbonate sodium chloride and sodium bicarbonate were mixed with an iron powder. The mixtures of the basic iron powder and pore-forming materials were compacted and sintered. The pore-formers were removed during or after the sintering process, leaving additional pores. The influence of the mixture composition on the compaction, the sintering, the green strength and the transverse rupture strength of the sintered parts were studied. The crucial role of the pore-former removal route was emphasized.

Keywords: highly porous sintered materials, pore-former, densification, green strength, transverse rupture strength

1. INTRODUCTION

Porous metallic materials are widely used as filters and sliding bearings in various areas of industry. For their manufacturing the powder metallurgy route is usually applied. This technology includes preparation of powder mixtures, compaction of green parts following by their sintering and often sizing. The compaction step can be excluded at manufacturing of some type of filters [1, 2]. The porosity of produced parts usually not exceeds 30-40%. Nowadays the demand for porous materials with much higher porosity has arisen. Such materials are being applied as sound and shock absorbing elements, anodes and cathodes of fuel cells and as medical implants. The increase in porosity is achieved by addition to the basic powder mixture some amount of pore-forming material for instance PMMA, ammonium bicarbonate, ammonium chloride, sodium chloride, sodium fluoride, carbamide etc. [3–5]. Removing of pore-former results the additional porosity witch level can be controlled by percentage of pore-former. The aim of present work is the investigation of influence of usage of different pore-formers on compaction behavior of powder mixtures and strength of obtained green and sintered parts. The work was carried out experimentally.

2. EXPERIMENTAL TECHNIQUE

As the basic material for manufacturing of porous samples the water atomized iron powder was used. The iron particles had a spongy form and the main fraction of 125-250 μm. The sodium chloride (NaCl), ammonium bicarbonate (NH_4HCO_3) and sodium bicarbonate ($NaHCO_3$) powders were used as pore-formers. The ammonium bicarbonate powder consisted of colorless particles irregular in shape with main fraction of 125-250 μm. The sodium chloride particles were white in color and crystalline in shape. The most representative fraction of this powder was 500-1000 μm. The particles of sodium bicarbonate had color and shape similar to sodium chloride ones, but they were much smaller in size. The main fraction of sodium bicarbonate particles was 63-125 μm. The fractions mentioned above were separated by sieving and used in all experiments. The basic iron powder and pore-forming powder were mixed manually for 15 minutes. The amount of pore-former was chosen as 50 % by volume. The theoretical densities of iron and pore-formers are sufficiently different: 7.874 g/cm^3 for the iron, 1.586 g/cm^3 for the ammonium bicarbonate, 2.165 g/cm^3 for sodium chloride and 2.515 g/cm^3 for sodium bicarbonate. The large difference in density can lead to segregation during mixing. To avoid segregation the small amount of gasoline (5% by weight) was added to all powder mixtures. The homogeneity of mixtures was controlled visually. The compaction of powders was studied by uniaxial pressing without lubrication in a cylindrical die with inner diameter of 16.8 mm. The masses of iron powder and powder mixtures were chosen to provide the height to diameter ratio of samples less then 1.15 over the whole range of compaction pressures. The mass of samples was 16.6 g for the iron powder, 12.2 g for iron and ammonium bicarbonate mixture, 16.6 g for iron and sodium chloride mixture and 17.0 g for iron and sodium bicarbonate mixture. The samples were compacted by pressure varying from 100 to 800 MPa with the step of 100 MPa. The force needed for ejection from the die was recorded for each sample. The ejection stress was calculated according to the equation

$$\sigma_{ej} = \frac{P_{ej}}{\pi \cdot d \cdot h}, \tag{1}$$

where P_{ej} – the ejection force and d, h – the diameter and the height of the sample. The density of compacts ρ was determined from the weight and size of the specimens. Then the relative density ρ_{rel} was calculated using relation

$$\rho_{rel} = \frac{\rho}{\rho_{th}}, \tag{2}$$

here ρ_{th} is theoretical density of powder mixture, determined according to equation

$$\rho_{th} = (1 - x) \cdot \rho_{Fe} + x \cdot \rho_{pf} \tag{3}$$

with x as pore-former volumetric content, ρ_{Fe} as theoretical density of iron and ρ_{pf} as theoretical density of pore-former. The samples for testing of bending strength were compacted in a rectangular die with the cavity size of 55.2 mm × 10.2 mm in plane. The samples weight was fixed for each mixture: 22 g for iron samples, 10.5 g for samples containing ammonium bicarbonate, 12.3 g for samples with sodium chloride and 14.0 g for samples with sodium bicarbonate. The height of samples has been decreased with increase of compaction pressure in the range of 4.38-5.62 mm for samples containing ammonium bicarbonate, 6.09-6.75 mm for samples with sodium chloride and 6.09-6.25 mm for samples with sodium bicarbonate. Some of these specimens were tested in the green state by three points bending loading. Other samples were sintered at temperature of 1150°C during 1 hour in reducing atmosphere of CO. Heating rate was maintained at 7°C/min. The intermediate dwell was carried out at temperature of 850°C during 30 minutes. The samples were firstly cooled with the furnace till 300°C and than in plain air up to room temperature.

During sintering ammonium bicarbonate was completely removed from the compacts due to decomposition above 60°C corresponding to reaction

$$NH_4HCO_3 \rightarrow NH_3 + H_2O + CO_2. \qquad (4)$$

Sodium bicarbonate has been decomposed to sodium oxide and carbon dioxide.

$$2NaHCO_3 \rightarrow Na_2O + H_2O + 2CO_2. \qquad (5)$$

Sodium oxide and sodium chloride has been washed out after sintering by holding in distilled water during 12 hours at temperature of 50°C. The attempts to wash out sodium chloride and sodium bicarbonate before sintering were unsuccessful due to drastic decrease of bonding ties between particles resulting crumbling of samples. The densities of sintered rectangular specimens were calculated based on their weight and size. The transverse rupture strength of sintered samples was determined by three point bending test.

3. RESULTS AND DISCUSSION

The densification curves for iron powder and its mixtures with pore-formers are shown in Fig. 1. The highest density at fixed pressure was observed for mixtures containing sodium chloride and the lowest one for iron powder. The densities of samples containing other pore-forming materials had intermediate values. This is a result of lower strength of pore-formers particles comparing to iron ones and their ability to fragmentation under pressure.

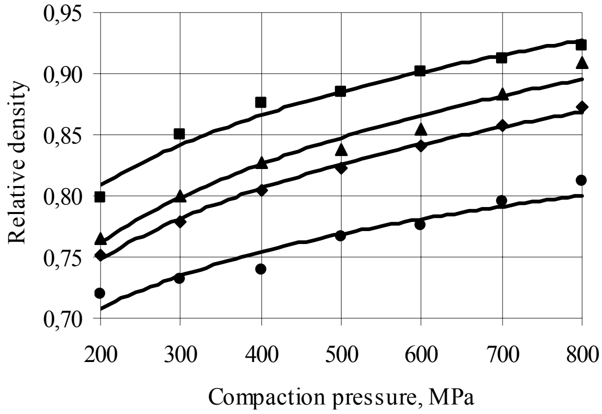

Fig. 1. The densification curves for the iron powder and the iron based powder mixtures:
● – Fe; ♦ – Fe–$NaHCO_3$; ▲ – Fe–NH_4HCO_3; ■ – Fe–NaCl

The variation of the ejection stresses with compaction pressure is shown in Fig. 2. The highest ejection stresses at a fixed compaction pressure were observed for the samples made of the iron powder. Ejection stresses for samples with pore-formers were much lower. This can be explained by action of pore-formers like a quasi lubricant due to their low shear strength. The best lubrication properties had sodium chloride powder. Ejection stress for iron samples was around 12% of compaction pressure whereas ejection stress for samples with pore-formers was only 3-4% of compaction pressure. All samples manufactured at specified conditions had no cracks. The influence of compaction pressure on green strength of powder samples is shown in Fig. 3. Strength of samples containing ammonium bicarbonate and sodium chloride was almost identical and essentially lower than strength of samples made of the pure iron powder. Large amount of pore-formers prevents the interlocking between the iron particles at pressing. At the same time, the strength of the mechanical contacts among particles of the pore-formers is much lower comparing to the strength of the contacts among iron particles. The green strength of samples containing sodium bicarbonate was negligibly low. Therefore these data are not presented here. The samples after sintering had a relative density of 0.400-0.475. The shrinkage of the samples made with ammonium bicarbonate and sodium bicarbonate and the swelling of the samples made with sodium chloride were observed. The variation of the bending strength of the sintered samples with the compaction pressure is shown in Fig. 4. As expected, the strength of the sintered compacts has been enhanced with the increase of the compaction pressure. The samples manufactured from iron and NH_4HCO_3 had the highest bending strength. The samples made with $NaHCO_3$ were much weaker. The samples produced with NaCl have the bending strength close to their strength in green state. This is a result of the incomplete removing of these pore-formers during sintering. The pore-formers or the products of their decomposition separate the iron particles, preventing their sintering. Thus, the ammonium bicarbonate is the best candidate among investigated pore-forming materials for production of high quality porous sintered parts.

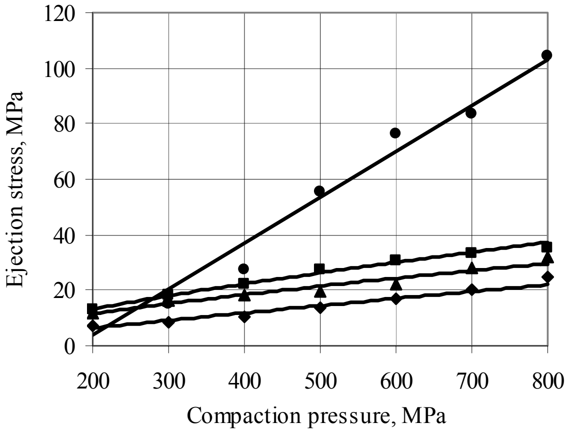

Fig. 2. The dependence of the ejection stresses on the compaction pressure for the iron powder and the iron based mixtures:
● – Fe; ■ – Fe–NaCl; ▲ – Fe–NH_4HCO_3; ♦ – Fe–$NaHCO_3$

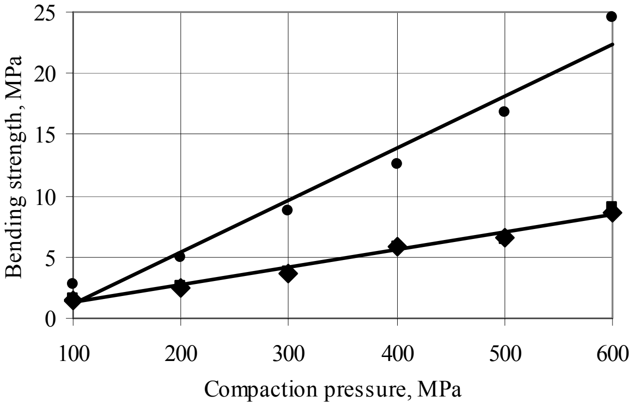

Fig. 3. The bending strength variation with the compaction pressure for the green samples made of the iron powder and the iron based powder mixtures:
● – Fe; ♦ – Fe–NH_4HCO_3; ■ – Fe– NaCl

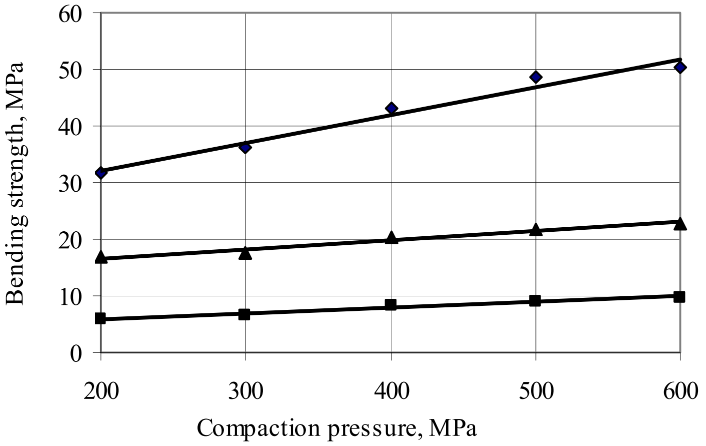

Fig. 4. The transverse rupture strength vs. the compaction pressure for the sintered samples made of the iron powder and the iron based powder mixtures:
♦ – Fe–NH_4HCO_3; ▲ – Fe–$NaHCO_3$; ■ – Fe– NaCl

4. CONCLUSIONS

The addition of pore-formers to the basic metallic material at production of highly porous materials has a crucial influence on densification, green strength and on transverse rupture strength of sintered parts. The relatively low shear strength of pore-former particles and their ability to easy fragmentation lead to increase in the densification efficiency comparing to pressing of basic metallic powder. The extent to which the densification is affected by pore-former depends on its type. For instance, in our case the sodium chloride had the largest influence on densification. The same peculiarities of pore-formers provide the sufficient lowering of the ejection stress in comparison to the basic metallic material. All investigated pore-formers had a similar influence on ejection stress of compacts. Pore-former particles are the obstacle for interlocking of the iron particles at compaction. This leads to the drastic decrease in green strength of compacts. The ammonium bicarbonate and sodium chloride have nearly same action on the fall in green strength whereas sodium bicarbonate reduces it nearly to zero. Very interesting is influence of pore-former type on sintering behavior. The shrinkage of compacts was observed when ammonium bicarbonate and sodium bicarbonate were used. Opposite to this, the usage of sodium chloride leads to swelling of compacts at sintering. During sintering ammonium bicarbonate decomposes to ammonia, carbon dioxide and water. These components have been fully removed at sintering. At the same time, the sodium bicarbonate decomposes to sodium oxide, carbon dioxide and water. The sodium oxide is preserved in compacts during sintering and thereafter can be removed by holding in hot water. Sodium chloride melts at 801°C and can be removed only after sintering as well. Thus, the sodium oxide and the sodium chloride are present in compacts during whole sintering cycle. They separate the iron particles from each other and block their sintering. As a result, the transverse rupture strength of sintered samples was low when sodium bicarbonate was used. The sintering had a minor contribution to the increase in strength of the samples pressed with sodium chloride. And the samples manufactured with ammonium bicarbonate had the highest transverse rupture strength after sintering. Thus, the way of pore-former removal has a crucial influence on the resulting strength of the sintered parts. The pore-former must be removed before the active phase of the sintering starts. In our case, only the ammonium bicarbonate suits to this requirement and can be recommended for usage in the production of high quality porous parts.

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