Quantitative image analysis in some iron powder metallurgy materials

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Abstract. The goal of the present paper is to analyze the porosity in some powder metallurgy (P/M) alloys by two different methods, using the conventional method and by image processing analysis method. Quantitative image processing is a new and useful tool which is able to determine the pore size, pore size distribution and porosity of parts obtained by P/M route. One important and significant disadvantage of P/M processing is the presence of porosity due to their activity as crack initiators and due to their presence as the stress distribution in compacts is nonhomogeneous in the cross-section. Two atomized iron powders obtained by powder metallurgy with particles of different sizes (< 45, 45-63, 63-100, 100-150, >150 µm) are the based materials studied in this paper. The analyzed powders were subjected to uniaxial pressing with 500 MPa as the applied pressure. The compacts disc dimensions obtained are \( \phi 8 \times 6 \) mm in dimensions. The compacts were sintered in a laboratory furnace at a temperature of 1.120 °C. The sintering time was 60 and 90 minutes. The sintered specimens obtained were analyzed regarding their density, microstructure and porosity. Porosity of iron-based P/M materials was measured using the conventional and image processing technique. A correlation between the experimental and software data analysis could be established.

1. Introduction
The industry with the most powder metallurgy’s applications is represented by the automotive and aerospace industry, where 90% of the total world powder production is represented by iron and alloy powder [1, 2]. In the P/M conventional technology, the products are made by the process of blending, compacting and sintering ferrous powder elemental or alloyed. The limitation of this technology is represented by the small voids present in the parts after sintering, known as pores or porosity. Porosity plays a major influence on the mechanical properties of the sintered parts obtained by P/M due to their actions as stress concentrators and as a consequence, low values for strength and toughness [3-9]. Due to the progress in the field of the materials science concerning the application of image analyse [10-16] it has become an important method used for the investigation of the porosity and particle size in sintered powder metallurgy materials. There are several image processing software available to analyse images, such as: ImageJ, MATLAB, Avizo, PerGeos, Image Pro, OMERO, ENVI, ICY and others. Image J [17] is one of free and useful image analysis software to analyze porosity in an image by applying filters and adjustments using Threshold binary function, the pore areas are recognized, so the software generates a separate image only with porosity and can calculate it as percentage. The goal of this paper is to study the porosity in some sintered powder metallurgy materials by applying an image processing software tool and to compare the obtained results with the values obtained by conventional method.

2. Experimental details
The specimens used in this study are represented by two comercial powders produced from atomized iron powders and iron-based pre-alloyed powder with Cu, Ni and Mo as raw material. In table 1 is presented the chemical composition of the powder samples.
Table 1. Chemical composition of analyzed powders.

| Powder type | Cu   | Mo  | Ni  | C   |
|-------------|------|-----|-----|-----|
| P1          | 0.10 | 0.03| 0.05| <0.01 |
| P2          | 1.50 | 0.50| 4.00| <0.01 |

The particle size distribution of analyzed powders was ranged in 45 and 150 µm. In Table 2 are presented the particle size distribution of analyzed powders. The metal powders were homogenized with a lubricant, respectively zinc stearate -1% and then compacted into cylindrical shape with the dimensions of 8 mm diameter x 6 mm height at a pressure of 500 MPa.

Table 2. Particle size distribution of analysed powders.

| Powder type | Particle size distribution, [%] |
|-------------|---------------------------------|
|             | <45 μm | 45-63 μm | 63-100 μm | 100-150 μm | >150 μm |
| P1          | 25     | 21       | 29        | 19         | 5       |
| P2          | 21.3   | 20.2     | 32.6      | 23         | 3.1     |

For both powders, zinc stearate was used as die lubricant during pressing. In P/M process lubricants are indispensable due to theirs action in reducing friction between powder particles and the die. Also, the lubricants are added because can improve significantly the powder’s compressibility. Afterward, the green compacts were subjected to sintering at temperature of 1120 °C for 60 and 90 minutes in a laboratory furnace. The microstructural characterization and porosity behaviour were performed on unetched specimens using an optical microscope OLYMPUS BX51M. Regarding the determination of porosity characteristics, 100x magnification for samples was used. Following sintering, the sintered density of the samples was measured using conventional method and by applying an image processing software tool.

3. Results and discussion

Concerning the density of the samples, green and sintered densities were calculated by using geometrical method.

Density of a green part ($\rho_g$) obtained by P/M route is calculated using the follow relation:

$$\rho_g = \frac{mg}{vg},$$

where $mg$ = mass of the sintered part, $vg$ = volume of the sintered part.

Density of a sintered part obtained by P/M route ($\rho_s$) is calculated using the follow relation:

$$\rho_s = \frac{ms}{vs},$$

where $ms$ = mass of the sintered part, $vs$ = volume of the sintered part.

The total porosity ($Pt$) of the sintered compact obtained by P/M route, in volume percent, is calculated using the follow equation:

$$Pt = 100 \left( 1 - \frac{\rho_s}{\rho_t} \right) \ [%],$$

where $\rho_s$, $\rho_g$, and $\rho_t$ are the sintered density, green density and theoretical density.

The green and sintered densities of the analyzed specimens calculated using the geometrical method are represented in Table 3.
### Table 3. Green and sintered densities of the analyzed specimens.

| Powder type | Green density, (g/cm$^3$), [$\rho_g$] | Sintered density, (g/cm$^3$), [$\rho_s$] |
|-------------|--------------------------------------|----------------------------------------|
|             | Sintered at 1120º C and 60 minutes    | Sintered at 1120 º C and 90 minutes     |
| P$_1$       | 6.78                                 | 6.83                                   |
| P$_2$       | 6.81                                 | 6.85                                   |

Concerning the microstructure characterization of analyzed samples, the images acquisition was carried out on unetched samples using a digital camera coupled to an optical microscope (Olympus BX51M), at 100x magnification. Examination of figures 1-4 shows the optical micrographs of the sintered specimen P$_1$ and P$_2$ with different sintering time, 60 and respectively, 90 minutes and the images obtained after rendering using Image J.

**Figure 1.** Microstructure of sample P$_1$ at 1120° C for 60 minutes: (a) initial image and (b) after software rendering.

**Figure 2.** Microstructure of sample P$_2$ at 1120° C for 60 minutes: (a) initial image and (b) after software rendering.
By using the quantitative image analysis method, a comparative study of porosity was performed. Also, using the Image J software it was possible to generate a 3D image of the surface for the analysed sintered samples and is presented in figures 5 and 6.
Figure 5. 3D image of the surface for the samples sintered at 1120° C for 60 minutes, obtained using Image J software: (a) P₁ and (b) P₂.

Figure 6. 3D image of the surface for the samples sintered at 1120° C for 90 minutes, obtained using Image J software: (a) P₁ and (b) P₂.

Concerning the porosity measurements, in table 4 are presented the values obtained using conventional method by density technique and by image analysis technique using an image processing software, Image J.

The results showed that the longer sintering time gives a higher sintered density values and smaller porosity content. This can be attributed to the diffusion enhancement, which leads to reduce pore size and close porosity. By increasing the sintering time, the porosity decreases [18-20]. Also, a low values in porosity in sintered state for sample P₂ is observed due to the alloying elements, Cu, Ni and Mo [21-26].
Table 4. Percentage porosity of analyzed alloys obtained from conventional method and from image analysis using Image J.

| Powder type | Porosity from conventional method, (%) | Porosity from image analysis, (%) |
|-------------|----------------------------------------|----------------------------------|
| P1          | Sintered at 1120°C for 60 minutes | 13.11                            |
|             | Sintered at 1120°C for 90 minutes | 13.00                            |
| P2          | Sintered at 1120°C for 60 minutes | 12.90                            |
|             | Sintered at 1120°C for 90 minutes | 12.40                            |
|             | Sintered at 1120°C for 60 minutes | 13.83                            |
|             | Sintered at 1120°C for 90 minutes | 13.40                            |
|             | Sintered at 1120°C for 60 minutes | 13.97                            |
|             | Sintered at 1120°C for 90 minutes | 13.94                            |

During sintering process, Cu forms a liquid phase, acting as a semi-permeable film around the Fe particles, which improves the bond between the powder particles, while Ni and Mo remain in a solid state, so a partial diffusion of Ni in Fe is obtained.

4. Conclusions

The porosity measurements of analyzed samples using image analysis software - Image J was correlated to the density of the sintered samples obtained by geometrical method. A correlation between higher density and a decreasing in porosity was established. The sample P2 had a lower porosity and a higher density. The measurements of porosity resulted from density technique are ranging from 13.11% to 13.00% for sample P1, and from 12.90% to 12.40% for sample P2. The porosity measurements resulted by using the Image J software are ranging from 13.83% to 13.40% for sample P1, and from 13.97% to 13.94% for sample P2. A correlation between the experimental and software data analysis was established.

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