

# POROSITY MEASUREMENTS AND ANALYSIS OF SINTERED AND THERMOCHEMICAL HEAT TREATED P/M COMPACTS

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## ABSTRACT

The objective of this paper was to examine the porosity in fluidized-bed carburizing on sintered alloys obtained by the powder metallurgy route using an image software analysis, and to compare the results with those obtained using the conventional porosity measuring technique. A material's porosity is a measurement of its vacancy percentage. The volume of empty space divided by the material's bulk volume, given as a percentage, determines the overall porosity of the material. The advancement of digital imaging and software has resulted in a novel and appropriate technique for figuring out the porosity of materials used in powder metallurgy.

KEYWORDS: powder metallurgy, fluidized bed carburizing, porosity, image software analysis

### **1. Introduction**

Powder metallurgy (P/M) is a flexible manufacturing technique that involves the production of components from raw metal powders. A great degree of customization may be achieved, material waste can be minimized, and the technology can manufacture the indicated forms [1].

Understanding and controlling the porosity of the finished product is essential for guarantee its mechanical integrity and functional efficiency [2-5].

In sintered P/M alloys, porosity needs to be adjusted and balanced. Even when a certain level of porosity may be appropriate or even desired for some applications, excessive porosity can also have an impact on mechanical properties and performance. Therefore, process parameter modifications and careful consideration are necessary to attain the proper density and porosity balance [6-8]. Understanding and controlling porosity is essential in applications where sintered P/M alloys are often used, such as filters, cutting tools, bearings, and automotive components [9-11].

Understanding porosity is essential for knowing the mechanical properties and overall performance of materials, as sintering, a critical process in powder metallurgy, involves heating and compacting metal powders to produce solid structures [12-15]. Alloying elements can be added to sintered P/M alloys to improve their characteristics [16-18]. The most often added alloying elements in powder metallurgy include phosphorus, manganese, nickel, copper, and molybdenum. Because copper melts at 1083 °C during the sintering process, it fills the holes, increasing the toughness and density [19-24]. Due to the creation of Ni-rich areas during solid state sintering, nickel (Ni) boosts the sintered density and improves hardness and strength by promoting local ductility [25-27]. The hardenability of molybdenum (Mo) reacts well [28, 29].

Applying heat, thermochemical treatments, or mechanical treatments is another method to enhance the characteristics of these alloys. A thermochemical process that offers great heat and mass transfer is fluidized bed carburizing [30-36].

Porosity has an important effect in determining the success rate of fluidized-bed carburizing, a procedure frequently used to improve the surface hardness and wear resistance of P/M compacts. In carburizing, a material's surface is coated with carbon to increase its hardness and resistance to wear. In order to achieve uniform and controlled carburization, the fluidized-bed variation of this technique involves suspending the components in a fluidized bed of carburizing material.

The microstructure of sintered alloys may now be assessed non-destructively and with high accuracy using image software analysis, which has become a powerful and efficient technique for characterizing and measuring porosity in these materials. There are a number of benefits regarding employing picture



software analysis for porosity measurement over more conventional techniques. With this method, big datasets may be evaluated quickly and automatically, leading to a deeper comprehension of the porosity distribution in the sintered alloy. It also reduces the subjectivity associated with manual approaches by providing accurate and quantitative results [37-39].

Obtaining high-resolution pictures of the microstructure of the sintered alloy is the initial stage in the image software analysis process. To get finegrained pictures of the material, methods like optical microscopy, scanning electron microscopy (SEM), or micro-computed tomography (micro-CT) are frequently used. The input for the analysis that follows is these photographs.

Once the images are obtained, sophisticated image analysis software is employed to process and quantify the porosity. The software utilizes algorithms to identify and segment the pores within the microstructure, distinguishing them from the dense matrix of the alloy. Parameters such as pore size, shape, and distribution can be precisely measured, providing valuable insights into the material's characteristics.

The quantitative data generated through image software analysis can be used to assess the impact of various processing parameters on porosity levels in sintered alloys. Also, it can investigate the influence of sintering temperature, powder particle size, and compaction pressure on porosity formation, aiding in the optimization of manufacturing processes to achieve the desired material properties.

Moreover, image software analysis is instrumental in quality control and assurance during the production of sintered alloys. By implementing automated analyses, manufacturers can efficiently monitor and ensure consistency in porosity levels across batches, reducing the defects and enhancing the overall reliability of the final product.

This work aims to investigate the porosity in a fluidized bed carburizing sintered P/M materials using an image software tool, and then to compare the results with those obtained using a conventional approach to establish a correlation.

### 2. Experimental procedure

In this study, the samples were made from the Fe powder alloyed with 1.755% nickel, 1.50% Cu and 0.50% Mo. The second powder were made from Fe with 4.00% Ni, 1.50% Cu and 0.50 % Mo. As a solid lubricant, 1% zinc stearate was used. Lubricants are essential to the P/M process and are often added to mixes as an additive to lessen friction between powder particles and between powder compacts and the die-wall. The lubricant considerably increases the powder's compressibility. The green compacts were

sintered for 60 minutes at 1150 °C in a laboratory furnace, after being obtained at 600 MPa pressure with a single die action. The obtained disc specimens have the dimensions of 8 6 mm. The samples were subjected to fluidized bed carburizing treatment at 930 °C during 90 minutes after cooling to room temperature the samples were. The sintered and carburized in fluidized bed density, porosity, and metallographic analysis were performed on the compacted samples. The sintered compacts' volumetric dimensional change was computed, and the specimens' overall porosity was assessed based on the variation between the measured and reference densities. One of the key advantages of image software analysis is its ability to perform threedimensional (3D) reconstructions of the porosity within the sintered alloy. This allows for a more accurate representation of the spatial distribution of pores, enabling researchers and engineers to identify trends and patterns that might not be apparent in traditional two-dimensional analyses. Understanding the 3D porosity distribution is critical for predicting the material's mechanical behavior and structural integrity [40].

## 3. Results and discussions

### 3.1. Density results

Table 1 shows the results from conventional technique measurements of the density in sintered and carburized in fluidized bed state.

Powder<br/>typeSintered<br/>density,<br/>(g/cm³), psCarburized in<br/>fluidized bed<br/>density, (g/cm³), pcP17.167.21P27.237.39

# **Table 1.** Sintered sintered and carburized influidized bed density for analysed specimens

### 3.2. Microstructure analysis

The microstructures of fluidized bed carburized samples were observed by optical microscopy (Olympus BX 51M) at 100x are represented in Figure 2. The optical micrographs of the carburized in fluidized bed specimen P1 exhibit larger and irregular holes when compared to the pores in the sample with the maximum density,  $P_2$ , as can be seen by examining Figure 2.



# 3.3. Porosity measurements

In Table 2 are presented the porosity measurements of P/M products calculated using the conventional method, using the density technique. Using the freeware application Fiji for image processing was another way to determine porosity. It may be used to identify porosity in images by applying filters, adjustments, and the threshold binary function. Using an optical microscope and a digital camera, pictures of non-etched materials were acquired. The application creates a separate picture with porosity by extrapolating the regions with pores using the Thresholding function, and it can compute the porosity in percents. Table 2 and Fig. 3 show the results of the porosity measurements of carburized in fluidized bed samples obtained by Fiji program. A few processing parameters, including temperature, sintering duration, powder size distribution, green and sintered density of compacts, and alloy type and quantity, influence porosity [6-8].

Table 2. The porosity of analysed alloys	in
carburized in fluidized bed state	

Powder type	Porosity from convential method, (%)	Porosity from image analysis, (%)
$P_1$	8.61	9.76
P <sub>2</sub>	7.82	8.44

The measurements of porosity resulted from density technique are ranging from 7.82% to 8.61%. The measurements of porosity resulted by using the Fiji software are ranging from 8.44 % for 7.21 g/cm<sup>3</sup> to 9.76% for 7.39 g/cm<sup>3</sup>. A correlation between higher density and a decreasing in porosity was established. The sample P<sub>2</sub> had a lower porosity. Also, Fiji software can generate a plot profile of the surface for the carburized in fluidized bed samples presented in Fig. 4.



Fig. 2. Micrographs of the non-etched carburized in fluidized bed samples a)  $P_1$ ; b)  $P_2$ 



Fig. 3. Processed images by using the image software for porosity measurements of carburized in fluidized bed samples: a) P<sub>1</sub>; b) P<sub>2</sub>



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*Fig. 4.* Plot profile of the surface for the carburized in fluidized bed samples, obtained using image software: a)  $P_1$ ; b)  $P_2$ 

### 4. Conclusions

The density values obtained by the geometrical technique were linked with the measures of porosity in the fluidized bed carburized condition of the investigated materials using an image analysis program.

It was found that there was a relationship between increasing density and reducing porosity. The porosity of sample  $P_2$  was lower.

There was a correlation found between the software data analysis and the experimental data analysis. The porosity measured from the image analysis technique was higher than the porosity measured from the density technique because the image analysis technique only accounts for open porosity; closed porosity is not taken into account. A decrease in porosity and a reduction in pore size was associated with an increase in sintered and carburizing in the samples' fluidized bed density.

The surface qualities and mechanical performance can be increased by optimizing the carburizing process settings by studying the porosity characteristics.

In conclusion, image software analysis has become an indispensable tool in the study of porosity in sintered alloys or subjected to a termochemical treatment, such as carburzing in fluidized bed, as presented in this paper. Its ability to provide accurate, quantitative, and three-dimensional insights into the material's microstructure empowers researchers and manufacturers to make informed decisions regarding process optimization, quality control, and the development of advanced materials with tailored properties.

Porosity measurements and analysis of fluidized-bed carburized P/M compacts are necessary

to ensure the reliability and high quality of the final product.

As technology continues to advance, image software analysis will play an increasingly significant role in advancing our understanding of porosity in sintered alloys and optimizing their performance in various applications.

### References

[1]. Jang G. B., Hur M. D., Kang S. S., A study on the development of a substitution process by powder metallurgy in automobile parts, J Mater Process Technol, p. 110-115, 2000.

[2]. Hamiuddin M., Correlation between mechanical properties and porosity of sintered iron and steels-a review, Powder Metall. Int. 18, p. 73-76, 1986.

[3]. Park J., Lee S., Kang S., Jeon J., Lee H., Kim H. –K., Choi H., Complex effects of alloy composition and porosity on the phase transformations and mechanical properties of powder metallurgy steels, Powder Technology, vol. 284, p. 459-466, 2015.

[4]. Lu Z., Wei D., Wei P., Liu H., Yan H., Yu S., Deng G., Contact fatigue performance and failure mechanisms of Fe-based small-module gears fabricated using powder metallurgy technique, Journal of Materials Research and Technology, vol. 26, p. 1412-1427, 2023.

[5]. Ehtemam-Haghighi S., Attar H., Ilya V. Okulov, Matthew S., Damon K., Microstructural evolution and mechanical properties of bulk and porous low-cost Ti-Mo-Fe alloys produced by powder metallurgy, Journal of Alloys and Compounds, vol. 853, 2021.

[6]. Narasimhan K. S., Sintering of powder mixtures and the growth of ferrous powder metallurgy, Materials Chemistry and Physics, vol. 67, p. 56-65, 2001.

[7]. Dyachkova N. L., Feldshtein E., Microstructures, Strength Characteristics and Wear Behavior of the Fe-based P/M Composites after Sintering or Infiltration with Cu-Sn Alloy, Journal of Materials Science & Technology, vol. 31, issue 12, p. 1226-1231, 2015.

[8]. Haynes R., A study of effect of porosity content on ductility of sintered metals, Powder Metall, vol. 20, p. 17-20, 1997.

[9]. Benkai L., Ding W., Li M., Zhang X., Tool wear behavior of alumina abrasive wheels during grinding FGH96 powder metallurgy nickel-based superalloy, Procedia CIRP, vol. 101, p. 182-185, 2021.



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[10]. Konstanty J. S., *Applications of powder metallurgy to cutting tools*, Woodhead Publishing Series in Metals and Surface Engineering, Advances in Powder Metallurgy, Woodhead Publishing, p. 555-585, 2013.

[11]. Bhadeshia H. K. D. H., *Steels for bearings*, Progress in Materials Science, vol. 57, issue 2, p. 268-435, 2012.

[12]. Kulkarni H., Dabhade V. V., Blais C., Analysis of machining green compacts of a sinter-hardenable powder metallurgy steel: A perspective of material removal mechanism, CIRP Journal of Manufacturing Science and Technology, vol.41, p. 430-445, 2023.

[13]. Piotrowski A., Biallas G., Influence of sintering temperature on pore morphology, Microstructure and Fatigue Behavior of Mo-Ni-Cu Alloyed Sintered Steel, Powder Metallurgy, vol. 41, no. 2, p. 109-114, 1998.

[14]. Djohari H., Martínez-Herrera J., Derby J., Transport mechanisms and densification during sintering: I. Viscous flow versus vacancy diffusion, Department of Chemical Engineering and Materials Science, MN55455-0132.

[15]. Chagnon F., Trudel Y., Effect of sintering parameters on mechanical properties of sinter hardened materials, Advances in P/M & Particulate Materials, NJ, vol. 2, p 14-97/14-106, 1997.

[16]. Maheswari N., Ghosh Chowdhury S., Hari Kumar K. C., Sankaran S., Influence of alloying elements on the microstructure evolution and mechanical properties in quenched and partitioned steels, Materials Science and Engineering: A; 600, p. 12-20, 2014.

[17]. Wu M. W., Tsao L. C., Shu G. J., Lin B. H., The effects of alloying elements and microstructure on the impact toughness of powder metal steels, Materials Science and Engineering: vol. A 538, p. 135-144.

[18]. Trivedi S., Mehta Y., Chandra K., Mishra P. S., Effect of carbon on the mechanical properties of powder-processed Fe-0.45 wt.% P alloys, Indian Academy of Sciences, vol. 35, part 4, p. 481-492, 2010.

[19]. Angel W. D., Tellez L., Alcala J. F., Martinez E., Cedeno V. F., Effect of copper on the mechanical properties of alloys formed by powder metallurgy, Materials and Design, vol. 58, p. 12-18, 2014.

**[20]. Marucci M. L., Hanejko F. G.**, *Effect of copper alloy addition method on the dimensional response of sintered Fe-Cu-C steels*, Advances in Powder Metallurgy and Particulate Materials, MPIF, p. 1-11, 2010.

[21]. Dong Y., Jun L., Wen J., Jie S., Kunyu Z., Effect of Cu addition on microstructure and mechanical properties of 15%Cr super martensitic stainless steel, Mater Des, vol. 41, p. 16-22, 2012.

[22]. Takaki S., Fujioka M., Aihara S., Nagataki Y., Yamashita Y., Sano N., Adachi Y., Nomura M., Yaguchi K., Effect of Copper on Tensile Properties and Grain-Refinement of Steel and its Relation to Precipitation Behavior, Mater Trans, vol. 45, p. 2239-2244, 2005.

[23]. Ramprabhu T., Sundar Sriram S., Boopathy K., Narasimhan K. S., Ramamurty U., Effect of copper addition on the fatigue life of low alloy C-Mo powder metallurgy steel, Metal Powder Report, vol. 66, issue 3, p. 28-34, 2011.

[24]. Yang X., Bai Y., Xu M., Guo S., Effect of Additive Cu-10Sn on Sintering Behavior and Wear Resistance of 316L Stainless Steel, Journal of Iron and Steel Research, International, vol. 20, issue 7, p. 84-88, 2013.

[25]. Chawla N., Babic D., Williams J. J., Polasik S. J., Effect of copper and nickel alloying additions on the tensile and fatigue behavior of sintered steels, Advances in powder metallurgy & particulate materials, part 5, 104, Princeton, NJ: MPIF, 2002.

[26]. Bernier F., Plamondon P., Bailon J. P., L.'Esperance G., Microstructural characterisation of nickel rich areas and their influence on endurance limit of sintered steel, Powder Metallurgy, vol. 54, issue 5, p. 559-565, 2011.

[27]. Sulowski M., Structure and mechanical properties of sintered Ni free structural parts, Powder Metallurgy, vol. 53, N. 2, p. 125-140, 2010.

[28]. Sanjay S. R., Milind M. S., Vikram V. D., Effect of molybdenum addition on the mechanical properties of sinter-forged Fe Cu C alloys, Journal of Alloys and Compounds 649, p. 988-995, 2015.

[29]. Li H., Cai Q., Li S., Xu H., Effects of Mo equivalent on the phase constituent, microstructure and compressive mechanical properties of Ti-Nb-Mo-Ta alloys prepared by powder metallurgy, Journal of Materials Research and Technology, vol. 16, p. 588-598, 2022.

[30]. Mansoorzadeh S., Ashrafizadeh F., The effect of thermochemical treatments on case properties and impact behaviour of Astaloy CrM, Surface and Coatings Technology, vol. 192, issues 2-3, p. 231-238, 2005.

[31]. Kazior J., Janczur C., Pieczonka T., Ploszczak J., *Thermochemical treatment of Fe-Cr-Mo alloys*, Surface and Coatings Technology, vol. 151-152, p. 333-337, 2002.

[32]. Radomyselsk I. D., Zhornyak A. F., Andreeva N. V., Negoda G. P., The pack carburizing of dense parts from iron powder, Powder metallurgy and metal ceramics, vol. 3, p. 204-211.
[33]. Askaria M., Khorsand H., Seyyed Aghamiric S. M., Influence of case hardening on wear resistance of a sintered low alloy steel, Journal of Alloys and Compounds, vol. 509, issue 24, p. 6800-6805, 2011.

[34]. Krauss G., *Microstructure residual stress and fatigue of carburized steels*, Proceedings of the Quenching and Carburizing, The Institute of Materials, p. 205-225, 1991.

[35]. Georgiev J., Pieczonka T., Stoytchev M., Teodosiev D., Wear resistance improvement of sintered structural parts by C7H7 surface carburizing, Surface and Coatings Technology, vol. 180-181, p. 90-96, 2004.

[36]. Sulowski M., How processing variables influence mechanical properties of PM Mn steels?, Powder Metallurgy Progress, vol.7, no. 2, 2007.

[37]. \*\*\*, Fiji software, https://fiji.sc/.

[38]. Marin M., Potecaşu F., Potecaşu O., Marin F. B., Image Analysis Software for Porosity Measurements in Some Powder Metallurgy Alloys, Advanced Materials Research, vol. 1143, p. 103-107, Trans Tech Publications, Ltd., 2017.

[39]. Dobrzanski L., Musztyfaga M., Actis Grande M., Rosso M., Computer aided determination of porosity in sintered steels, Archives of Materials Science and Engineering, vol. 38, no. 2., p. 103-11, 2009.

[40]. Dobrzanski L. A., Musztyfaga-Staszuk M., Luckos A., *The comparison of computer methods for porosity evaluation in sintered constructional steels*, Journal of Achievements in Materials and Manufacturing Engineering, vol. 61, no. 2, p. 395-402, 2013.