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# Combined carbon content assessment method for powder metallurgy

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## ABSTRACT

**Purpose:** Powder metallurgy (PM) lacks a clear method to analyse the combined carbon content based on metallography visualisation, and this article describes the creation of such a method for powder materials.

**Design/methodology/approach:** Different methods are used to analyse combined carbon within metallurgical samples, and the hardness of components within the automotive industry is related to this question.

**Findings:** The main aim of this paper is to determine if optical microscopy provides a reliable means to assess the combined carbon content.

**Research limitations/implications:** For checking these items, the Optical Microscope will be used, density, hardness of sinter material, and particle size laser analysis of powder for creating the observed compact, and SEM microscope.

**Practical implications:** This investigation provides standardised rules that can be implemented within any material laboratory.

**Originality/value:** The analysis of powder particle size, hardness test, density check, and the investigation of the structure of powder element are presented.

**Keywords:** Powder materials, Combined carbon content, Porosity, Particle size analyse

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## METHODOLOGY OF RESEARCH

## 1. Introduction

The carbon content must be assessed in powder metallurgy to calculate production costs. Manufacturing companies monitor costs by consuming carbon supplied as loose powder and assessing the carbon content of specific components after production. An appropriate method to check the carbon content after compacting and sintering processes is required for such calculations to be reliable. Literature often refers to standards and tests to check the carbon content of classical alloys, but powder materials have, in addition, the porosity structure; therefore, this publication takes into account an adapted method to check the carbon content under the microscope.

Carbon is a key element in powder metallurgy and is the basic alloying element in PM sintered steel. Carbon powders are also used as the carbon source to produce hard metals. Carbon allotropes such as graphite are mixed into iron or iron-based powders with lubricant powders and sometimes other additives during the preparation of powder mixes. Graphite is almost fully dissolved during sintering into the iron matrix [1].

### 1.1. Microstructure

Combined carbon is the portion of carbon in iron and steel that is chemically united in the form of carbides [2].

Pearlite is a mixture of alternately arranged ferrite and cementite plates resulting from the eutectoid transformation occurring at 723°C, which contains 0.8% C [4]. An example of a pearlitic microstructure is presented in Figure 1, using its trade name ASC100.29+0.75%C etched with 1% Nital.

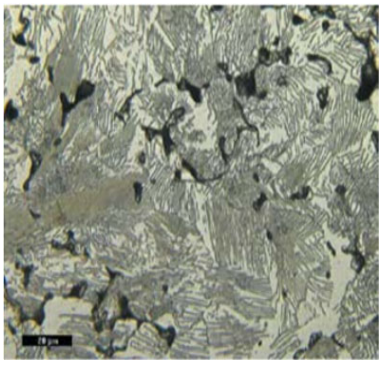


Fig. 1. Example of pearlitic microstructure [3]

Ferrite is a solid carbon solution in α iron, crystallising in a spatially-centred cubic lattice (A2). This solution has a very low carbon content of 0.008% in the buffer and 0.0218% when treated at 723°C. It is soft and ductile, and ferromagnetic up to the Curie temperature of 768°C, after which it becomes paramagnetic [4]. An example of a ferritic microstructure is presented in Figure 2, using the trade name ASC100.29 (atomised iron powder grade), which has been etched with 1% Nital. This etchant attacks the grain boundaries and reveals the grain structure of the ferrite material [3].

The carbon content of a sintered material can be estimated on a metallographic section from the area fraction of pearlite, where 100% pearlite is equivalent to approximately 0.8% carbon. Carbon dissolves rapidly in iron at the temperatures normally used during the sintering of carbon steels, and it is unusual to see uncombined carbon in sintered steels [3].

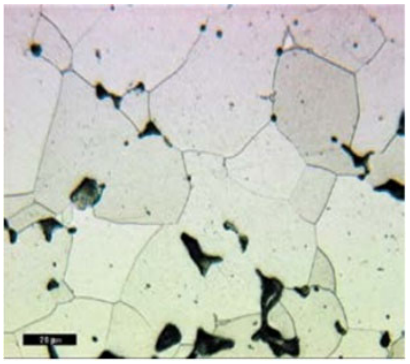


Fig. 2. Example of ferrite microstructure [3]

### 1.2. Porosity

Porosity is characterised as the pore volume ratio to a porous body's nominal volume. It is stated as a percentage or a decimal. [5].

Porosity is very application-based rather than a property for which a single material fits all needs, so the appropriate porosity depends primarily on how a part will be used. When parts must have high strength, porosity can be undesirable, but there are many applications where powder metallurgy can provide precise control over porosity. In general, a density of 4-5 g/cm$^3$ is considered high porosity. When you reach 5 to 6.5 grams, porosity becomes a balancing act [6].

Pore size measurements must be conducted on the raw materials used for powder metallurgy processes to ensure that the end products yield desirable mechanical properties. The strength, weight, hardness, and permeability of metal alloys produced from powder minerals depend upon the porosity of the raw constituents [7].

Apart from the non-trivial geometrical texture of composites in general, the carbon content of composites is particularly prone to various types of geometric imperfections and relatively high porosity arising directly from the fabrication process. This is characterised by the thermal decomposition and transformation of an initial polymeric precursor into a pyrolytic carbon matrix through carbonisation, re-impregnation, and graphitisation. The major contribution to the porosity, which may exceed 30% of the overall volume, is crimp voids and delamination cracks, which are usually classified as inter-tow voids. Intra-tow voids represented by pores and transverse cracks also contribute [8].

### 1.3. Sample preparation

The presence of carbonaceous contaminants, either on the surface of the sample or in the pores, interferes with determining the carbon content. Combined carbon may be

determined by metallographic comparison with reference microstructures for ferrite-pearlite.

The list below shows the literature steps for sample preparation for a visual microscopy check:

a) Heat the specimen to 1000 ± 20°F (540 ± 10°C) for 15-30 minutes in the air. Remove the specimen from the heat and allow it to cool. A longer time may be required for parts with a higher or lower density (< 6.6 g/cm$^3$) [9].
b) Reduce the test specimen to a size appropriate for the analysis instrument. Take care to avoid overheating, oxidation, contamination, or loss of any free graphite particles during machining or subsequent handling.
c) Put the samples in the mounting press and fill 1/3 of resin with glass fibre and then 2/3 with bakelite. Process the sample under high pressure during the heating and cooling cycles.
d) Removed the damaged or deformed surface material by grinding.
e) Open all pores to show the true area fraction of porosity, and remove scratches by polishing for 15 minutes.
f) Keep the preparation times as short as possible because extended preparation times may damage the sample, and the true pore and edge roundness will not be determined.
g) Use Nital Fe 1 100 ml - 95% Ethyl alcohol, 1 ml concentrated $HNO_3$. The time for the etching step is 10-60 seconds. The amount of pearlite and ferrite is used to estimate the carbon content.
h) Measure the total carbon content of the fully-prepared test specimen using the procedure described in ASTM E1019 [3].

### 1.4. Relation between combined carbon content and hardness

The cause that makes it difficult to maintain the production process of the intended carbon content is that the starting Fe powders should be reduced to 0.1-0.5%, which burns out during the sintering of coal. A controlled atmosphere cannot be checked with the carbon in the sinter during sintering initiation because decarburisation and carburisation processes take place during this process, making it difficult to regulate the carbon content [10].

Based on the observation of the materials, it was found that the share of pores in individual layers of gradient materials decreased upon increasing the carbon concentration in individual layers. Increasing the carbon concentration decreases the sintering temperature in all layers, while the density of pressed and sintered samples increases with increasing sintering temperature. The hardness of pressed and sintered samples increases upon increasing the coal concentration and sintering temperatures [11].

## 2. Materials and experiments

The research method is based on the proper preparation of a sample for metallographic examination using an optical microscope. The test aims to define a method to check the carbon content of metallurgical powders by specifying the type of material, appropriate sample preparation, and microscopic analysis.

The tested material is FC0208-40 with a surface finish. This material consists of iron, copper (1.5-3.9 wt%), and carbon (0.6-0.9 wt%). A visual check was carried out using SEM, and hardness and density measurements were made according to MPIF 42 [12].

### 2.1. Microscope

To analyse particle shape and size of the powder, SEM was used, and the chemical composition of the powder was checked by EDS analysis. For a direct combined carbon content check, an optical microscope was used. A Zeiss Axio Imager M1m microscope was used to image the microstructures, and observations were made in the bright field view technique. The determination of the percentages of individual structure components was performed using ImageJ software.

### 2.2. Hardness

The Rockwell Superficial 3JS was used for hardness measurements.

### 2.3. Density

The density of the samples was determined per MPIF 43 [13]. An analytical balance, a container with water, and a vacuum system were used for density checks.

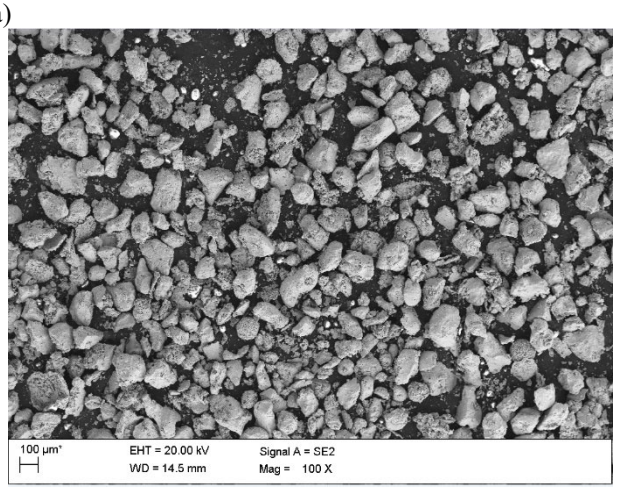
## 3. Results

### 3.1. SEM analysis

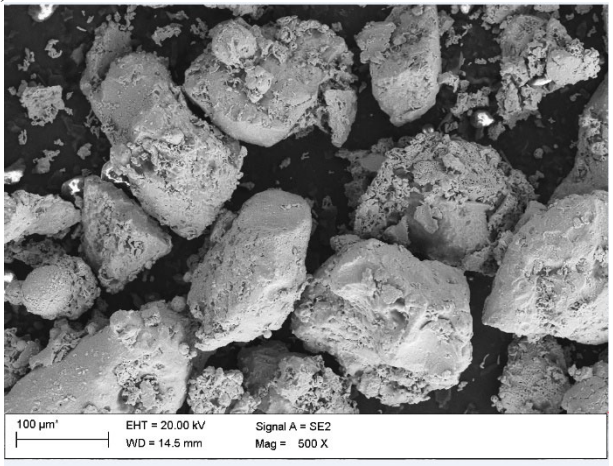
The versatile chemistry of carbon allows it to create endless chains, sheets, and three-dimensional structures. Carbon (particularly in the sp$^2$ hybridisation state) can create a virtually unlimited number of structures and forms materials usually referred to as graphenic carbon materials. Graphenic carbonaceous solids are made up of carbon atoms bonded through sp$^2$ hybridisation. Such materials include graphite, activated carbon, chars, cokes, glass-like carbons, carbon blacks, and also popular nanostructures of graphene, carbon nanotubes (CNTs), and nanofibers (CNFs) [14].

Figure 3 shows the SEM analysis of powder particles with magnifications of 100x, 500x, and 1000x.

a)




b)




b)

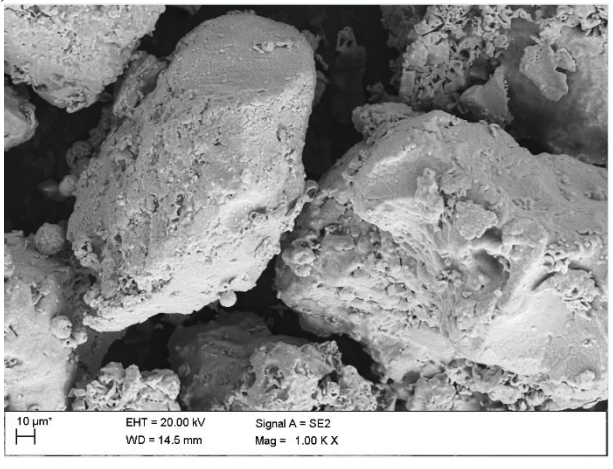



Fig. 3. SEM analysis of FC-0208-40 powder particles; magnification: a) 100x, b) 500x, c) 1000x

EDS analysis was also performed to characterise the elemental content of samples. For this purpose, the areas marked in Figures 4-6 were analysed. The detailed chemical composition is presented in Table 1 and Table 2.

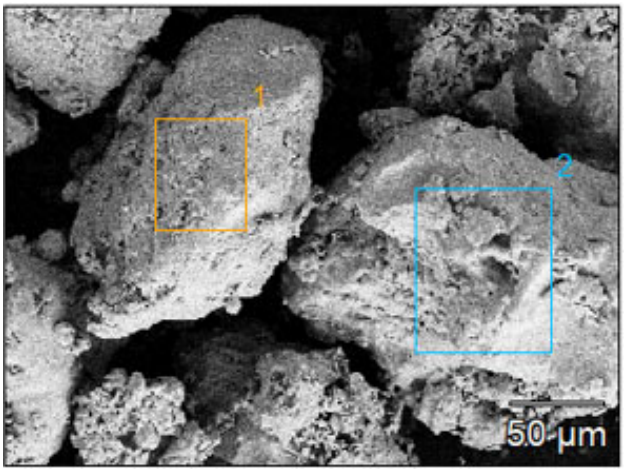



Fig. 4. Area for EDS analysis

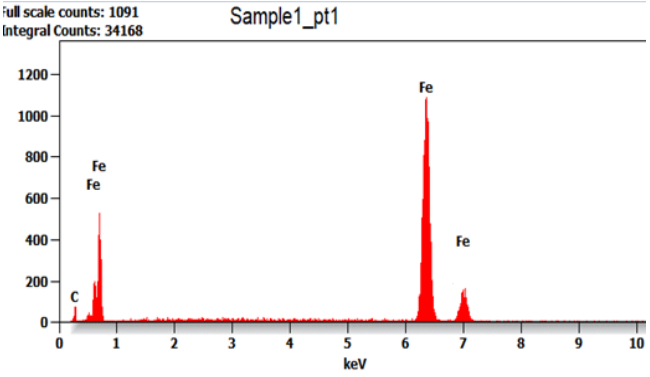



Fig. 5. EDS results for area 1

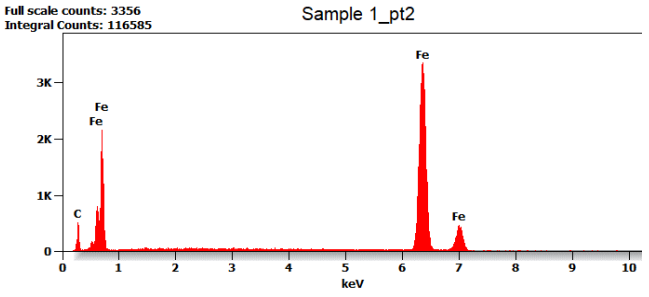



Fig. 6. EDS results for area 2

Table 1.
Weight %

| | C | Fe |
|---|---|---|
| Check 1 | 3.0 | 97.0 |
| Check 2 | 6.3 | 93.7 |

Table 2.
Atom %

| | C | Fe |
|---|---|---|
| Check 1 | 12.4 | 87.6 |
| Check 2 | 23.7 | 76.3 |

The analysis data:

- Image Name: Sample 1;
- Image Resolution: 512 by 384;
- Image Pixel Size: 0.65 µm;
- Acc. Voltage: 20.0 kV;
- Magnification: 1000.

### 3.2. Hardness

The hardness of the samples was determined per MPIF 42 [12], and the results are presented in Table 3. It was found that both samples were within the required hardness range, 55-90 HRF.

Table 3.
Hardness Results

| Sample | Hardness, HRF |
|---|---|
| 1 | 78.0-82.5 |

### 3.3. Density

The samples showed an average density of 6.37 g/cm$^3$. It confirms the table data for the density of FC-0208-40 according to MPIF 35 [15].

### 3.4. Experiment A

A section from each sample was removed and prepared per ASTM E3 [16], and the results are presented in Figure 7 and Figure 8.

The sample preparation steps:
Step 1: Grind to 220 grit.
Step 2: Polish with 9-micron Diamond spray.
Step 3: Polish with 3 micron Diamond spray.
Step 4: Polish with 1 micron Diamond spray.
Step 5: Etching by 2-3% Nital.

Combined carbon by way of the visual estimate was 0.4-0.5%. Within the microstructure were found ferrite and pearlite.

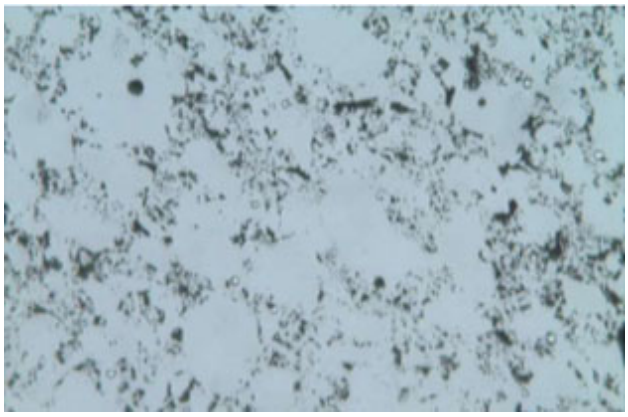


Fig. 7. Sample A; non-etched microstructure (magnification 100x)

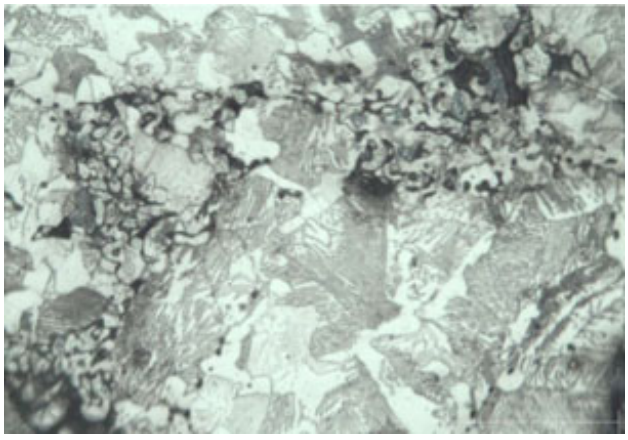


Fig. 8. Sample A. Etched microstructure (magnification 500x)

### 3.5. Experiment B

A section from each sample was removed and prepared per ASTM E3 [16], and the results are presented in the photographs below (Figs. 9-11).

The sample preparation steps:
Step 1: Fine grinding 15-6 µm.
Step 2: Plane grinding of samples with a hardness of 150-1000 HV.
Step 3: Polish with 3-micron diamond spray.
Step 4: Polish with 1-micron diamond spray.
Step 5: Etching by 2-3% Nital.

Before each step, the sample was washed with tap water and then isopropanol.

Combined carbon by way of visual estimate was 0.72%. Within the microstructure were found ferrite and pearlite.

Fig. 9. Sample B. Non-etched and oxides and pores (magnification 100x)

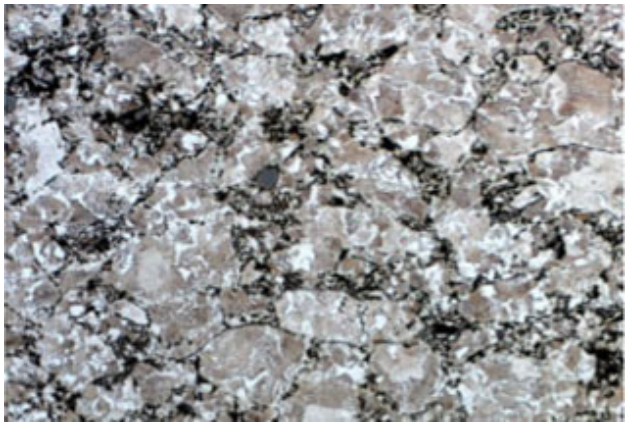


Fig.10. Sample B. Etched 2% Nital, oxides + pores, pearlite + ferrite (magnification 100x)

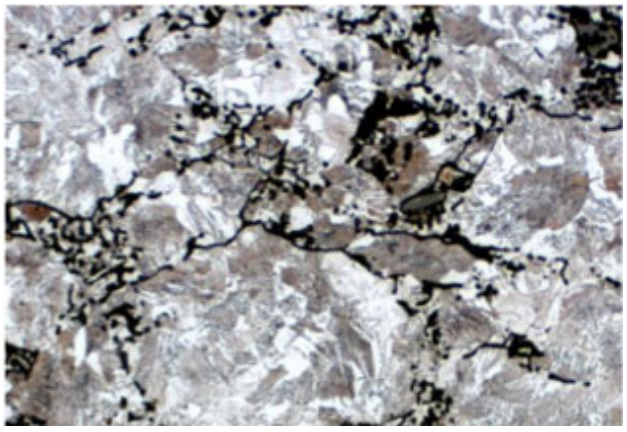


Fig. 11. Sample B. Etched 2% Nital, oxides + pores, pearlite + ferrite (magnification 500x)

### 3.6. Calculations

To calculate the amount of carbon, the steps below need to be performed:

1) Determine the percentage of pores and oxides;
2) Determine the relative surface of ferrite and pearlite;
3) Determine the percentage of ferrite and pearlite without taking into account the pores and oxides;
4) Estimate the carbon content in the tested samples assuming that there is 0.77% carbon in the pearlite.

The real data for FC-0208-40, experiment B, are presented in Table 4.

Table 4.
The porosity percentage

| SAMPLE B | The porosity percentage |
|---|---|
| Etched, oxides+pores, % | 12.1% |
| Ferryt, % | 6.2% |
| Pearlite, % | 81.7% |
| Combined carbon content, %mass | 0.72% |
| Assumption: Sum of ferrite and pearlite equals 100% (after subtracting the percentage of pores and oxides) [17] | |

## 4. Conclusions

This study aimed to determine the percentage of pearlite to estimate the carbon concentration in tested samples, assuming that 100% pearlite represents 0.77% carbon.

This experiment showed the importance of the sample preparation method for metallographic tests because both specimens were made of the same material, FC-0208-40. The required density should be 6.3 g/cm³ according to the standards, as confirmed by the density test. The hardness of the samples was in the 55-90 HRF range. The grain size complied with the laboratory requirements with a standard deviation below 5%.

This study demonstrated the importance of the magnification used to check the structure. Experience has confirmed that to better define the ferrite and pearlite content necessary for determining the carbon content; samples must be etched with 5% Nital. The observation should be made at 100x magnification. The optical microscopes and cameras used for the tests were equivalent, despite having different manufacturers. This was confirmed by experiments A and B, which showed different carbon contents for the same sintered material. Experiment A from 0.4-0.5%, while experiment B showed about 0.7%, which was similar between contractors.

This method should be used in powder metallurgy to facilitate often unnecessary disputes between powder producers, the factories producing composites, and direct customers. It provides a simple and practical way to analyse combined carbon content that is accessible to anyone analysing the above-mentioned issues.

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