Investigation of Powder Metallurgy Material Using Laser Ultrasonic Method

Yordan N. MIRCHEV¹, Pavel N. IVANOV², Elena B. CHEREPETSKAYA², Mitko M. MIHOVSKI¹, Vadim A. KOVTUN³

¹ Institute of Mechanics at the Bulgarian Academy of Sciences, Sofia, Bulgaria
e-mails: mirchev@imbm.bas.bg, nmtd@abv.bg.

² National University of Science and Technology “MISiS”, Moscow, Russia
e-mails: pavelivanov@mail.ru, echerepetskaya@mail.ru

³ Gomel Branch University of Civil Protection of the Ministry for Emergency Situations of the Republic of Belarus; Gomel, Belarus
e-mail: vadmikov@ya.ru

Abstract
The paper presents the results of complex studies of powder metallurgical materials based on iron powder. The study of the materials was carried out with the application of the methods of metallography and ultrasonic using innovative equipment with laser excitation of ultrasonic waves.

Keywords: powder metallurgical materials, laser ultrasonic technique.

1. Introduction
The technology of powder metallurgy allows producing materials with unique physical and mechanical characteristics. By choosing the composition of the powder, the porosity, and various heat treatment are realized according to the requirements in initial conditions of the assignment. One of the difficulties in preparing the products is the even distribution of the porosity, especially in thin-walled products, such as bearings. The reduced porosity leads to difficulties in lubricating the bearing, which raises its temperature. The high porosity reduces the strength characteristics of the elements.
In the work, an experimental study of the distribution of porosity in model samples with conventional and innovative ultrasonic and metallographic methods is carried out.

2. Testing object
The experiment was performed on powder metallurgical materials based on iron powder type NC-100-24 from the company Hogines, alloyed with copper powder. Table 1 shows the modes for preparation of the samples, where P is the porosity, T - sintering temperature, τ - sintering duration, TO - heat treatment after quenching-annealing at T =550 °C for 120 min.

<table>
<thead>
<tr>
<th>Sample №</th>
<th>P, %</th>
<th>Cu, %</th>
<th>T, °C</th>
<th>τ, min</th>
<th>HB annealing</th>
<th>TO</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 (81)</td>
<td>25</td>
<td>5</td>
<td>1150</td>
<td>120</td>
<td>62</td>
<td>-</td>
</tr>
<tr>
<td>2 (85)</td>
<td>25</td>
<td>5</td>
<td>1150</td>
<td>240</td>
<td>71</td>
<td>+</td>
</tr>
<tr>
<td>3 (44)</td>
<td>15</td>
<td>5</td>
<td>1150</td>
<td>30</td>
<td>85</td>
<td>+</td>
</tr>
<tr>
<td>4 (87)</td>
<td>25</td>
<td>5</td>
<td>1150</td>
<td>240</td>
<td>45</td>
<td>+</td>
</tr>
<tr>
<td>5 (80)</td>
<td>25</td>
<td>5</td>
<td>1150</td>
<td>120</td>
<td>65</td>
<td>+</td>
</tr>
<tr>
<td>6 (111)</td>
<td>15</td>
<td>10</td>
<td>1150</td>
<td>240</td>
<td>87</td>
<td>+</td>
</tr>
</tbody>
</table>
The samples have dimensions of 100x10x3 mm. They are obtained by the method of single pressing in the direction of the size of 3 mm. After sintering and thermal annealing, the samples are ground until roughness of $Ra \approx 2 \mu m$. Table 1 also introduces additional data obtained when estimating HB hardness by the Brinell method, as an average of 5 measurements.


3.1. Laser ultrasonic equipment

Laser ultrasonic technique has a number of advantages over conventional ultrasonic technique [1]. One of the significant advantages is the more precise determination of the attenuation and velocities of the ultrasonic waves propagating in the material. It is used in cases where precise determination of the acoustic characteristics of the material is necessary in order to obtain the physical-mechanical characteristics of the materials [2, 3], as well as determination of the changes in the structure of the tested materials [3].

Laser ultrasonic equipment UDL-2M, manufactured by LINKS 2000 - Moscow, is used for the research. It is designed for precise measurements of the velocity of longitudinal ultrasonic waves in solids by echo-pulse measurement technique. A block diagram of the equipment is shown in Fig.1.

![Fig. 1. block diagram of the equipment UDL-2M.](image)

The block diagram includes the following blocks: 1 - optical-electronic unit, 2-optical cable and optical-acoustic sensor (PLU-6P-02), 4 - test sample, 5 - personal computer. An electronically controlled mechanical manipulator is used to move the sensor relative to the sample. The optoelectronic unit is differentiated on the basis of a pulsed Nd: YAG laser with a high frequency of the generated pulses [4, 5]. The laser radiation enters the sensor 3 by means of an optical cable. The probe 3 includes: an optoacoustic generator, an acoustic waveguide and a piezoelectric receiver in which both the generated ultrasonic pulse and the pulses which are reflected by imperfections and/or the bottom of the testing sample enter. A diagram of the probe is shown in Fig.2.

A laser pulse enters in the wide-frequency optoacoustic transducer via an optical cable between the laser and the probe, which generates longitudinal ultrasonic waves by thermal action. The optoacoustic generator is a plate with flat parallel walls made of plastic, absorbing the laser pulse. It generates an acoustic signal in the sample and is at the same time a receiver that transmits the signals through the waveguide to the piezoelectric transducer. The laser ultrasound system operates in the frequency range 0.1 $\div$ 15 MHz. The diameter of the ultrasonic beam is 3 $\div$ 4 mm. The pulse repetition frequency is 0.5KHz. The relative error in measuring the velocity of ultrasonic waves is 0.05 %. The operating frequency of the piezoelectric plate is 0.1 $\div$ 6 MHz. The depth resolution is 0.1 mm. Sensitivity to minimal defect is 0.3mm. The duration of the generating acoustic pulse is of the order of 70 ns. The recommended compressive force applied to the probe is 0.1 MPa. The dead zone of the probe when working in a steel environment is of the order of 50 $\mu$m.
Fig. 2. Probe PLU-6P-02 in working conditions. 1 - acoustic waveguide, 2 - Optical acoustic generator, 3 - sample, 4 - imperfection, 5 - optical cable, 6 - piezoelectric transducer, 7 - data acquisition and processing system (PC), 8 - ultrasonic pulse, 9 - laser.

3.2. Conventional ultrasonic testing
Traditional measurements of longitudinal ultrasonic wave propagation velocities were performed by the 5 MHz frequency transmission method using an Epoch 600 ultrasonic flaw detector with a set of sensors. A strong inhomogeneity of the mean velocity was recorded in the measurements along the length of the samples.

3.3. Porosity test
The porosity was determined by weight method by determining the volume and weight of the samples. The average porosity of the materials is determined.

3.4. Structural testing
The structure of the materials was determined using a standard metallographic microscope and Phenom electron microscope, ProX modification.

4. Results

4.1. Results of metallographic examination of the samples
The microstructure of some of the samples is given in fig. 3. The photo of Fig. 3a shows the particles of the starting powder. NC 100-24 The following photos show the structures of some of the samples to be examined, and the magnification is given in brackets. The structure is heterogeneous. The presence of pores and the formation of copper particles around the iron dust particles is observed.

a) matrix powder (x100)  b) sample № 1 (x100)  c) sample № 1 (x2000)
4.2. Results of electron microscopy examination of a sample № 3.
The studies were performed on 4 areas of the surface of sample № 3, presented in Fig.4. A relatively uniform distribution of copper powder particles in the iron matrix is observed. The distribution of the pores is relatively even. In different areas the iron content varies from 81.3 to 86.3%, the copper content - from 13.7 to 18.7%. Traces of carbon in percentages from 6 to 13% were also found, which is an accompanying additive for the case of section preparation.

Fig.4 Image of a composite material based on iron powder with a Phenom ProX electron microscope.

4.3. Distribution of the velocity of propagation of longitudinal ultrasonic waves by height (3 mm) of the samples.
A typical image obtained from the ultrasonic laser system is presented in Fig.5. The scan was performed along the length of the sample, marked with the X axis. The pulses from the sample surface, from the bottom and from the first bottom and secondary reflected signal are presented in blue, orange and green, respectively. Unevenness of the structure along the height of the sample is observed. Uneven stripes are observed in the image due to the significant change in the average velocity (height of the sample) presented in the direction of the scan. To the left of the image is the time of arrival of the pulse in µs.
Fig. 5 Typical B-image from scanning with the laser ultrasound system of a composite material based on iron powder.

Fig. 6 shows the distribution of the velocities in the direction of movement of the sensor for the tested samples.

![Graphs showing velocity distribution](image)

**Fig. 6 Longitudinal wave propagation velocities distribution along the length of the specimens.**

Table 2 shows the measured maximum and minimum values of the longitudinal velocities $C_l$, the average porosity $P$ and the average velocity $C$, determined by measured 10 values along the length of the sample.

<table>
<thead>
<tr>
<th>Sample №</th>
<th>$C_l$ max, m/s</th>
<th>$C_l$ min, m/s</th>
<th>$\Delta C_l$, m/s</th>
<th>$P$, %</th>
<th>$C$, m/s</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4500</td>
<td>3700</td>
<td>800</td>
<td>25</td>
<td>4100</td>
</tr>
<tr>
<td>2</td>
<td>4780</td>
<td>4250</td>
<td>400</td>
<td>25</td>
<td>4300</td>
</tr>
<tr>
<td>4</td>
<td>4780</td>
<td>3800</td>
<td>520</td>
<td>25</td>
<td>4390</td>
</tr>
<tr>
<td>5</td>
<td>4500</td>
<td>4100</td>
<td>980</td>
<td>25</td>
<td>4550</td>
</tr>
<tr>
<td>6</td>
<td>4780</td>
<td>4100</td>
<td>650</td>
<td>15</td>
<td>4420</td>
</tr>
</tbody>
</table>
The dependence of the velocity of propagation of longitudinal waves on the porosity is presented in the literature and written as follow:

\[ C = C_0 (1 - \alpha \cdot p) \]  

where \( C_0 \approx 5850 \text{ m/s} \) is the velocity of propagation of the ultrasonic wave in a material of ARMC iron, \( \alpha \) - coefficient taking into account the structure and technology for the manufacture of the material.

5. Conclusion

The used experimental equipment complex allows mechanical movement of the sensor on the surface of the controlled object and obtaining the velocity distribution along the length of the sample in the direction of sound. Both imperfections and changes in the density of the material are registered.

The obtained results are especially useful for the practice in order to optimize the pressing processes to obtain products with minimal changes in porosity. The measured average values of the velocity in the separate parts of the object give information about the strength characteristics.

Acknowledgements

This research was funded by NUST MISIS Competitiveness Program, grant No. K2-2019-004.

References