Experimental research about the obtaining of the iron synthesized composite materials

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Abstract. The sintered pieces of metallic powders show a particular physical and chemical characteristics, determined both by the composition and the existing phases structure, as well as by the size, shape and distribution of the grains. This paper aims to show the peculiarities of sintering production of a class of composite materials with the iron matrix and their characterization regarding the physico-mechanical properties obtained after sintering. Thus, in order to obtain parts with advanced compactness, the process of double pressing and double sintering is applied. The first pressing is performed with moderate pressures (3000 daN/cm²) and sintering at 800°C with a range of about 30 minutes. The second pressing is performed at high pressures (6000÷7000 daN/cm²) and is followed by the final sintering in normal mode.

For fused metal sintered metal composites, the most important mechanical property is hardness. This is a function of the bonding forces between the particles, the density and the strength of the material at the test site and can be determined by the Brinell method.

1. Introduction
Composite materials are a category of nonconventional materials made of a metallic or non-metallic matrix reinforced by "sintering" with various particles of rough surfaces, fibers and other elements [1]. These are materials with anisotropic properties, consisting of several components, the use of which allows the use of their best characteristics such that they have final properties generally superior to the components, which constitute [2-4].

Powder metallurgy as a process for the manufacture of the composite materials is industrially utilized, characterized by the use of powdered raw materials, their agglomeration, their pressing into predetermined shapes and sizes, followed by the strengthening of the bonding between the particles by sintering, ensuring thus the final properties. It can be said that at present there is no branch of the industry that does not use or cannot use sintered powdered products under advantageous conditions [5, 6].

Powder metallurgy can be applied in two situations, namely:
- In order to obtain achievable products and other processes;
- To make products impossible to obtain by other processes.

Pieces made of sintered composite materials through powder metallurgy have a number of technical and economic advantages, including [7, 8]:
- Very high coefficient of use of the raw material;
- High precision products, corresponding to Class H9 and even H7;
- The possibility of replacing expensive or deficient materials;
- High productivity;
- Universal base machine.

Limiting the field of use of the powder metallurgy in machine building is due to causes that are more in line with current state of the technics. Thus, can be:
- The high cost of raw materials;
- The high cost of specific product-specific devices;
- The limits imposed on the shape of the piece by the pressing process used;
- Limits imposed on the dimensions of the parts, determined by the capacity of the operating equipment.

2. Current technologies for the production of sintered metal matrix materials

At present, the sintered composite materials are made based on a technological process consisting of a succession of main operations [9, 10]:

- Preparation of materials (drying of non-metallic powders, for the removal of moisture; sizing of powders, in order to obtain the desired granulometric fractions);
- Dosage of raw materials (metallic powders, non-metallic powders, additives and lubricants, if applicable);
- Homogenizing the powder mixture with the desired composition;
- Molding of homogenised materials (with or without pressure in molds);
- Sintering of parts resulting from training;
- Final processing.

2.1. Raw materials

The raw materials for the sintered composite materials are divided into:

- Metal powders;
- Non-metal powders;
- Lubricants and additives.

2.1.1. Metal powders. Metal powders are a set of granules of less than 1 mm in diameter, of pure metals, alloys, metallic chemical compounds or mechanical mixtures of a several components. Metal powders may be non-alloyed, partially alloyed or fully alloyed.

Characteristics of metallic powders, which affect the properties and quality of sintered composite materials, refers to [10-13]:

- chemical properties (chemical composition, alloying or impurity content, corrosion resistance in various media, chemical reactivity under different conditions such as pyrophoricity of powders, oxidation resistance under different storage conditions, etc.);
- physical properties (particle size and particle size distribution, particle shape, internal particle structure, surface particle quality, particle specific particle size, particle micro-particle density, actual particle density, etc.);
- technological properties (apparent filling density, Compressed density, flowability, filling coefficient, compressibility, stability of the shape of the blank after compaction, ie edge strength, etc.).

Studies made in the field of powder metallurgy have shown that the properties and quality of the sintered products and their manufacturing technology depend to a large extent on the method of manufacturing the powdered metal powders used as starting material, respectively, on the chemical, physical and technological properties of these powders.

Thus, it can change sensitively the presetability of powder blends and porosity achievable by pressing as well as the intergranular diffusion conditions, the degree of homogeneity of the alloy and pseudo alloy structure formed during sintering and the sintering contraction, respectively, the final porosity and mechanical, physical properties and technological aspects of sintered materials.

An ideal metal powder used in powder metallurgy must:

- Have a high compressibility;
• Give the product a high mechanical strength;
• Exhibit good flow properties;
• Exhibit minimal dimensional variations during sintering;
• Provide high mechanical strengths and high elongation to sintered parts.

2.1.2. Non-metal powders. In the category of non-metallic powders used for the production of sintered composite materials are included [14]:
• Sliding additions (graphite, C, molybdenum, MoS$_2$);
• Friction additions (quartz, SiO$_2$, alumina, Al$_2$O$_3$, asbestos, mullite, 3Al$_2$O$_3$ 2SiO$_2$, silicon carbide, SiC);
• Filling materials (baritone, BaSO$_4$).

2.1.3. Lubricants and additives. Lubricants are chemicals used in powder metallurgy to ensure uniform pressure distribution during powder Compressed and ease ejection (outlet) of the tablets from the mold [15, 16].

Lubricants are used in the range of 0.2÷1% and have the following effects when pressing metal powders:
• A slight increase in powder compressibility;
• Reduction of ejection pressure;
• Elimination of physical defects in the tablets (cracks, density unevenness etc.);
• Reducing the friction between the powder particles, between the punches and the mold, between the powders and the mold, between the tablet and the mold walls during the ejection;
• Removing mold grip.

Additives (binders), added to metallic powders at a rate of 1÷5%, increase the adhesion between the powder particles and thereby cohesion of the tablet, thus eliminating physical defects in compact material. Lubricants and binders may be present in the form of a powder, a liquid, aqueous solutions or organic solvents.

The side effects of using lubricants and binders are:
• The occurrence of residual carbon, which affects the physical, mechanical and chemical properties of the sintered parts;
• The occurrence of oxides, which influences the technological properties of the sintered parts (machinability, dimensional constancy);
• The occurrence of solid or liquid residues in the sintering furnace-cooling zone.

2.2. Sintering of the metallic powders pieces
Sintering is the process of welding, densification and recrystallization by thermal activation of particulate agglomerates in the absence or in the presence of a liquid phase. The metal powder tablet is strengthened during sintering by forming stable and stable bindings between the granules. The surface, volume, gap, or interstitial diffusion phenomena, at the grain boundary, provide for the formation of bonds between granules.

The variable sizes that occur during the sintering process of the metal powder tablets are [16]:
• Material transportation (evaporation, condensation, surface diffusion, volume diffusion, diffusion at the granule boundary);
• Sintering temperature;
• Heating and cooling speed, sintering time;
• The sintering atmosphere;
• Sinter activation additives;
• Surface condition and particle size.

It should be emphasized that the choice of the sintering variables is largely determined by economic factors. Metal sintering practically starts at half the melting temperature.
2.3. Iron sintering

Iron sintering temperatures are between 1050°C and 1200°C. When sintering the pure technical iron up to 600°C, the lubricant sludge removal from the press, the reduction of superficial oxide layers and the restoration of the crystalline network takes place, thus canceling the existing internal stresses in the powder granules or caused by the operation pressing [17-22].

In the temperature range from 600°C to 850°C, the formation of bonding bridges between the granules takes place, which results in increased tensile strength of the sintered iron.

Over 906°C the transformation Feα in Feγ takes place. Under this transformation point, autodiffusion is very fast (about 400 times faster than in the γ phase). During the other stages of sintering, in addition to the progressive spheronization of the pores, there is a faster growth of the granules.

In order to obtain parts with advanced compaction, the double pressing and double sintering process is applied. The first pressing is performed with moderate pressures (3000 daN/cm²) and sintering at 800°C with a range of about 30 minutes. The second pressing is performed at high pressures (6000÷7000 daN/cm²) and is followed by the final sintering in normal mode.

Alloys in the iron-carbon system are used in powder metallurgy for two purposes:

- for obtaining carbide parts with wear and high toughness while the interior remains ductile;
- for the production of materials with high tensile strength.

This ultimate goal is achieved by alloying the iron with the carbon in the whole mass of the workpiece by mixing graphite with iron powder, since iron and graphite are not soluble in solid state.

The sintering of the iron-graphite system is characterized by the diffusion of a part of graphite, with the formation of a metal structure with a perlite structure. Carburation occurs through the mechanisms shown in figure 1.

The chemical reactions that occur are difficult to control, resulting in variable loads from one load to another, especially for products with a content greater than 3% graphite.

The formation of the perlite-graphite structure imposed on anti-friction materials is very difficult to achieve because at the sintering temperature of the components (1050°C÷1100°C), austenite is able to dissolve more carbon than is necessary for the formation of the perlite structure (figure 2).

In order to avoid the formation of secondary cement in the structure, the technological variant of the mixing of iron powder and copper powder graphite was proposed in a certain percentage.

The factors influencing the sintering of iron-graphite mixtures are: the quality of the iron powder; the quality of graphite and the composition of the sintering atmosphere.
It is necessary for the Fe powder to have a loss in low H₂, this loss must be known in order to accurately determine the amount of graphite added. The added graphite quality is generally defined by its particle size, fine particle powders having a higher reactivity. Atmosphere and sintering conditions have a great influence on the properties of sintered iron-graphite mixtures, with the risk of carbonation.

It is very difficult to determine exactly whether a sintering atmosphere is carburated, decarburated or neutral because in the sintering furnace its composition changes with temperature, furnace load and evaporation from sintered materials. The sintering temperature and the combined carbon influence the dimensions of the sintered iron-graphite parts [17-22].

3. Experiments on the obtaining and characterization of metal sintered metallic materials with iron matrix

3.1. Elaboration of recipes

Recipes chosen for sintered materials with iron matrix are shown in table 1.

| Table 1. Recipes of metallic materials sintered with the iron matrix. |
|-------------------------|---------|---------|---------|---------|
| Type of material | Fe  | Cu  | Ni  | Graphite | SiO₂ | SiC |
| Fe-1          | 68  | 13  | 7   | 6       | 8    | -   |
| Fe-2          | 68  | 13  | 5   | 6       | -    | 8   |

3.2. Preparing the raw materials

The preparation of the raw materials has the effect of removing the impurities and moisture, factors that have a negative influence on all subsequent operations of the technological flow, with repercussions on the properties of the sintered material.

Simultaneously with the removal of impurities, the granulometric dosing is carried out by the powdering operation applied to the powders. Water removal is achieved by keeping hygroscopic powders in the oven at about 180°C.

The raw materials and granulometric fractions used in the prepared recipes were as follows:

- Iron powder, type PJV 3-160/GOST 9849-89, < 0.2mm;
- Copper powder, grade IV/STAS 10283-81, < 0.2mm;
- Nickel powder, carbonyl type/STAS 9707-74, < 0.071mm;
- Graphite powder, grade II, dry grade 3, STAS 1903-65, 0.1÷0.3mm;
- Powder as black SiC, Turda type < 0.12mm;
- Quartz sand powder, Miocani type, STAS 5923-76, 0.12mm.

3.3. Homogenisation of mixtures

The powders prepared according to the preceding paragraph were dosed according to the formulations in table 1 and the resulting mixtures were homogenized in order to achieve homogeneous mixtures, which ultimately ensure good reproducibility of the physico-mechanical properties of the sintered material.

For homogenization, cylindrical laboratory oscillators with inclined axis were used, the homogenization time being 3 hours.

3.4. Sample formation

For the formation of samples from the Fe-1 and Fe-2 mixtures, a cylindrical die having a surface area of 2 cm² was used. Compressed pressures ranged from 5000÷6000daN/cm². The values obtained for "raw" density are shown in table 2.
Table 2. The values for "raw" density for iron-based sintered metallic materials.

| No. | Type of material | Compressed pressure (daN/cm²) p | Compressed density (g/cm³) |
|-----|------------------|---------------------------------|---------------------------|
| 1   | Fe-1             | 5000                            | 5.12                      |
| 2   | Fe-1             | 6000                            | 5.37                      |
| 3   | Fe-2             | 5000                            | 5.51                      |
| 4   | Fe-2             | 6000                            | 5.63                      |

3.5. Sample sintering
The sintering of Fe-1 and Fe-2 samples was carried out in the SAFED tunnel furnace with exo-type protection under the following conditions: sintering temperature: 1100°C; gas flow rate “exo”: 4Nm³/h; furnace speed: 0.6 m/h.

3.6. Testing of sintered samples
A. Density. For sintered samples, density was determined by weighing on the METTLER analytical balance and measuring, with a micrometer, the volume calculation. The values obtained for the density of the metal sintered metal with the iron matrix are shown in table 3.
B. Hardness. The hardness of the sintered samples was determined using the WOLPERT dyemeter, using the following test regime: penetration diameter: 2.5mm; test force: 62.5daN; duration of the test: 2 min. The values obtained are shown in table 3.

Table 3. The values of density and hardness of sintered samples from Fe-1 and Fe-2 materials.

| Type of material | Compressed pressure (daN/cm²) | Sintering temperature (°C) | Sintered density (g/cm³) | Sintered hardness (HB) 5 |
|------------------|-------------------------------|-----------------------------|--------------------------|--------------------------|
| Fe-1             | 5000                          | 1100                        | 4.68                     | 8.6                      |
| Fe-1             | 6000                          | 1100                        | 4.72                     | 62.0                     |
| Fe-2             | 5000                          | 1100                        | 5.02                     | 80.3                     |
| Fe-2             | 6000                          | 1100                        | 5.10                     | 105.8                    |

4. Conclusions
The experiments we performed showed the influence of the technological parameters and composition of the materials on the physico-mechanical and tribological characteristics obtained.

In the compositions with the iron base (matrix), we were looking to achieve great results, using fewer constituents in the recipe, so that the technological process is as simple as possible. At the same time, regarding this category of materials, we studied the influence of friction additions such as SiO₂ and SiC.

In experimental research, in addition to the determination of physico-mechanical quantities such as sinter density and Brinell hardness, we also performed an electron microscopy study on sintered compositions. Therefore, we highlighted the basic metal matrix and the distribution of the other constituents.

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