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Determing the Compensating Action of Copper After Sintering of Powder Metallurgical Structural Steels

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***Abstract*— This paper studies the dimensional changes during sintering of steel powders structural materials alloying with a 2,5 percents copper compound. Studied were three types of steel powders – NC 100.24; SC100.26 and ASC 100.29. Carbon concentration in the test pieces is between 0,2 and 0,8 percents, and their density between 6,2 and 7,0 grams per cubic centimeter. Presented are graphical relationships of the dimensional changes depending on the carbon concentration in the tested samples and their density.**

Keywords*— *carbon concentration, density, dimensional changes, iron powder, permeability.

I. INTRODUCTION

With the advent of Industry 4.0 in recent years, there is a distinct trend towards increasing the degree of digitization, connectivity, autonomy and intelligence of production processes. Their efficiency, productivity and accuracy are constantly being improved, while at the same time shortening the response times and the processing cycles. Innovative and energy-saving technological processes are taking bigger share in the production of goods and supplies from all branches of industry [1] – [9]. The development of non-conventional technological processes, in addition to their energy-saving advantages, make it possible to obtain new materials that are impossible to obtain by conventional technologies. In this regard, the contribution of powder metallurgy is significant [10] – [14].

The powder metallurgical technology allows obtaining up-to-date construction materials from which details can be made, the chemical composition and mechanical properties of which cannot be achieved by conventional methods. Such elements as copper, phosphorus, tin, etc., which are considered harmful impurities in conventional steels, are widely used in powder metallurgy [11], [15], [16].

Copper is an element that is not used in conventional metallurgy, but has a number of features that favor its use in powder metallurgy. The more important of them are:

- can be obtained in the form of powder by different technologies;
- the obtained copper particles can be of different shapes;
- has easily recoverable oxides;
- high plasticity, which facilitates pressing;
- lower melting temperature than iron, which causes sintering in the presence of a liquid phase, etc. [11], [12], [15].

Along with the mentioned advantages of copper, its addition in powder metallurgical samples leads to significant changes in their linear dimensions [11], [15].

In this regard, the aim of the present study is to track the change in linear dimensions after sintering of iron carbon powder metallurgical structural materials alloyed with 2.5% copper.

II. MATERIALS AND METHODS

In the research process, powder metallurgical samples of three types of iron powders produced by the company "Höganäs" - Sweden - NC100.24, SC 100.26 and ASC 100.29 were developed.

One of the most widespread iron powders obtained by the reduction method in the practice of powder metallurgy production is NC 100.24. The compactability of this brand of iron powder is very good, and thanks to the spongy structure of the particles, their formability is very high. The raw (after pressing) and final (after sintering)

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strengths of parts from these powders are very high, and the hydrogen concentration in them is low.

The SC 100.26 type iron powder has the best compactability of all the sponge iron powders produced by the company "Höganäs" using the reduction method. This makes it particularly suitable to produce parts with a single pressing, to which there are requirements for higher density. Like the powder of the previous type, SC 100.26 also has a high raw strength, but a slightly higher density. It is used in the production of details that will later be subjected to chemical-thermal treatment.

Iron powder ASC 100.29 is a representative of the group of water-dispersed powders. Now it is the highest quality iron powder produced by the company "Höganäs". It is very clean. It can be compacted with great results, because the particles are almost spherical in shape. This allows, after a single pressing, to achieve a density of 7.2÷7.3g/cm^3. These powders are particularly suitable to produce structural products with high density, as well as for products with certain magnetic characteristics.

The mixture for the samples, in addition to iron powder, contains 0.2÷1.0% standard graphite powders brand UF - 4, with the carbon concentration in the range of 96÷97% and 2.5% electrolytic copper with a particle size of 63µm. The ingredients of the charge are placed in a non-metallic cylinder of a drum mixer with intersecting axes of rotation and 0.8% of the coating substance "Kenolube" is added. The resulting batch is homogenized for 20 minutes.

After mixing, the ingredients of a sample are weighed on electronic scales with accuracy of 0.001g. The mass is determined depending on the pressing effort and the desired density [13], [14]. The weighed powders are placed in the pressing matrix, the dimensions of which are 0.03÷0.07% larger than the dimensions of the sample bodies - ø25 h=20mm. The pressing was carried out bilaterally on a fast-acting press with an effort of 200÷800 MPa. This allows obtaining samples with a density in the interval 6.2÷7.0g/cm^3.

The samples were sintered in a laboratory horizontal muffle furnace with a "Carbolitte" ceramic tube in the presence of dissociated ammonia - NH3. – fig.1.

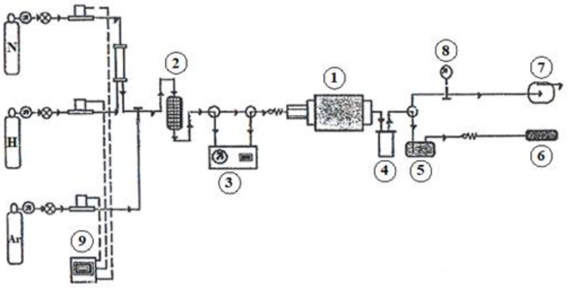


Fig.1. The sintering system;
1 – furnace; 2 - molecular dryer – sieve; 3 – hygrometer SHAW; 4 - separator plasticizer; 5 - oil valve; 6 – lighter; 7 - rotary vacuum pump; 8 - pressure gauge; 9 - computer (software MGF&PC).

The quantitative ratio of nitrogen and hydrogen in the working space of the crucible is 75%H_2 and 25%N_2. The sintering was carried out at a temperature of 1150°C for 1 h controlling the speed of the incoming gas and its dew point.

To prevent oxidation of the samples during the sintering process, closed ladles and backfill (75%Al_2O_3+15%FeMn+10%C) were used. The backfill helps to limit to a minimum the mass loss of the samples because of the oxidation of the iron powders at the high sintering temperatures.

The heating of the samples takes place in two stages:

- First stage - The samples are heated to 800ºC and kept at this temperature in order to separate the plasticizer - fig.1 position 4.
- Second stage - Heating continues at a rate of 10ºC/min until isothermal sintering temperature - 1150°C.

The dimensional changes of the studied samples - Δl, is determined in percentages as a ratio of the dimensions after pressing and after sintering.

III. Results and discussion

The experimental results for the three investigated brands of iron powders with different concentrations of carbon in them and density in the range of 6.2÷7.0g/cm^3 are presented in table 1, and their graphic interpretation in fig.2÷4, respectively, for an iron matrix of powders NC100.24; SC 100.26 and ASC 100.29.

Table 1 Experimental Results

A

ρ, g/cm^3	Δ l, %				
	Fe	***Fe + C***			
		C, %			
		0,2	***0,4***	***0,6***	***0,8***
Iron powder type NC 100.24					
6,2	-0,14	-0,17	-0,13	-0,04	-0,01
6,4	-0,11	-0,15	-0,07	-0,01	+0,02
6,6	-0,09	-0,11	-0,02	+0,01	+0,05
6,8	-0,08	-0,09	0,00	+0,05	+0,07
7,0	-0,07	-0,03	+0,02	+0,08	+0,09
Iron powder type SC 100.26					
6,2	-0,14	-0,11	+0,01	+0,05	+0,08
6,4	-0,12	-0,10	0,00	+0,04	+0,08
6,6	-0,09	-0,09	0.00	+0,04	+0,07
6,8	-0,07	-0,07	-0,01	+0,03	+0,06
7,0	-0,06	-0,05	-0,01	+0,03	+0,06
Iron powder type ASC 100.29					
6,2	-0,21	-0,17	-0,15	-0,7	-0,02
6,4	-0,18	-0,16	-0,13	-0,05	-0,01
6,6	-0,16	-0,14	-0,10	-0,05	-0,01
6,8	-0,13	-0,10	-0,07	-0,03	0,00
7,0	-0,09	-0,08	-0,05	0,00	+0,01

B

ρ, g/cm^3	Δ l, %			
	Fe + C + 2,5% Cu			
	C, %			
	0,2	***0,4***	***0,6***	***0,8***
Iron powder type NC 100.24				
6,2	+0,22	+0,21	+0,18	+0,16
6,4	+0,22	+0,19	+0,17	+0,13

6,6	+0,21	+0,19	+0,15	+0,11
6,8	+0,21	0,18	+0,13	+0,09
7,0	+0,20	+0,16	+0,11	+0,08
Iron powder type SC 100.26				
6,2	+0,27	+0,25	+0,24	+0,19
6,4	+0,26	+0,24	+0,23	+0,18
6,6	+0,26	+0,23	+0,22	+0,18
6,8	+0,25	+0,22	+0,21	+0,17
7,0	+0,25	+0,21	+0,19	+0,17
Iron powder type ASC 100.29				
6,2	+0,50	+0,46	+0,37	+0,30
6,4	+0,49	+0,44	+0,35	+0,29
6,6	+0,47	+0,39	+0,32	+0,27
6,8	+0,45	+0,35	+0,30	+0,24
7,0	+0,42	+0,31	+0,29	+0,22

The values obtained during the measurements show that in the samples of pure iron after sintering, the change in the linear dimensions has a negative sign, therefore, a shrinkage of the samples is observed, which is of the order of -0.14÷-0.20% in samples with density 6.20g/cm^3. As the density of the samples increases to 7.00 g/cm^3, the internal porosity of the blanks decreases, and hence the possibility that the blanks undergo a large change in their dimensions during the coalescence of the powder particles. Therefore, in samples with high density, the change in dimensions decreases almost twice and is within the limits of -0.07÷-0.09%.

The addition of carbon to the matrix leads to a decrease in the compressibility of the blanks, but despite this, at a carbon concentration of 0.2%, the tendency for the dimensional change to be in the negative part remains. For samples containing 0.4%C and a density of 6.80g/cm^3 made of iron powder NC100.24, practically no changes in the linear dimensions of the blanks are observed. The same was found for samples of SC 100.26 iron powder with a density of 6.40÷6.60 g/cm^3. For samples made because of iron powder ASC 100.29, linear changes are not observed at a density of 7.00 g/cm3 and 0.6%C, and for samples with 0.8%C, the dimensions of the samples do not change at a density of 6.80 g/cm^3. Therefore, we can draw the conclusion that at low carbon concentrations - 0.2÷0.6%C, powder Fe-C samples decrease in size, like those made of pure iron. When the carbon concentration increases above 0.6%, however, a change in the linear dimensions of the samples is found in a positive direction - towards their growth. The concentration interval in which the powder metallurgical structural details of the Fe-C system will not suffer linear changes, or they will be insignificant - 0.4÷0.6%C.

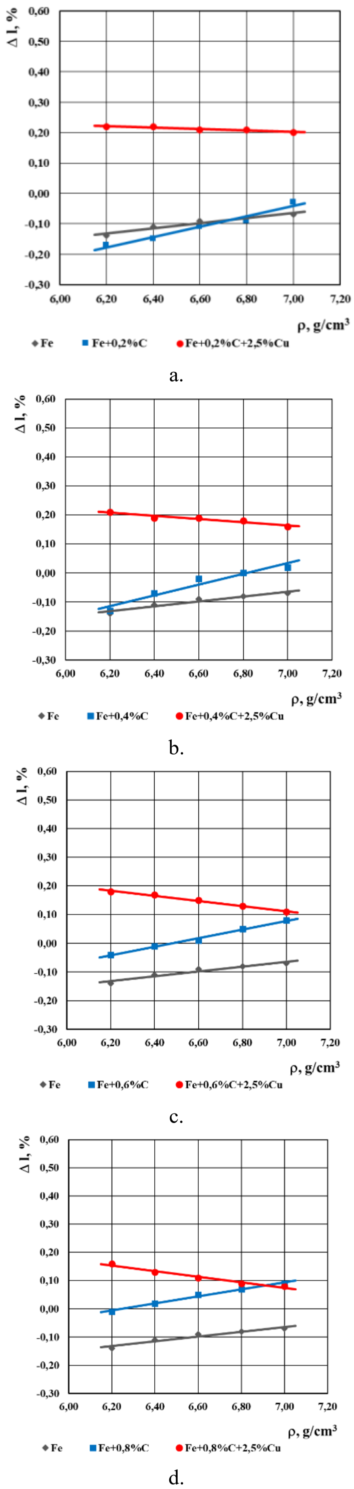
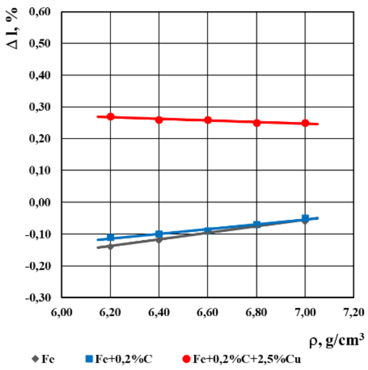
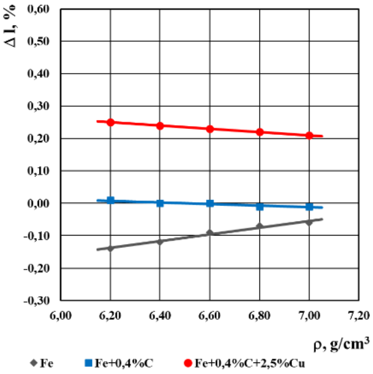


Fig.2. Distribution of values for linear changes depending on density and the carbon concentration of NC 100.24 iron powder samples: a – 0,2%C; b – 0,4%C; c – 0,6%C; d – 0,8%C



a.



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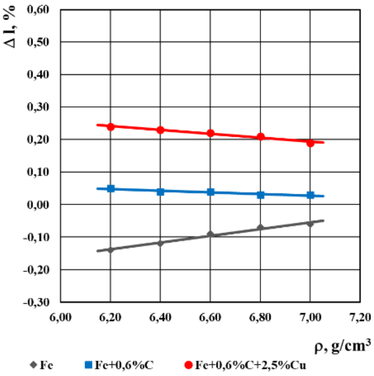
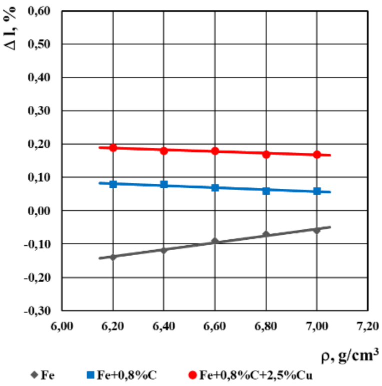
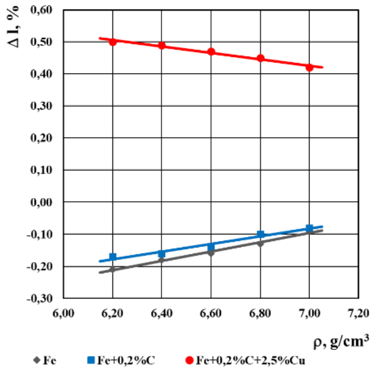


c.



d.

Fig.3. Distribution of the values for the linear changes depending on the density and the carbon concentration of SC 100.26 iron powder samples; a – 0,2%C; b – 0,4%C; c – 0,6%C; d – 0,8%C



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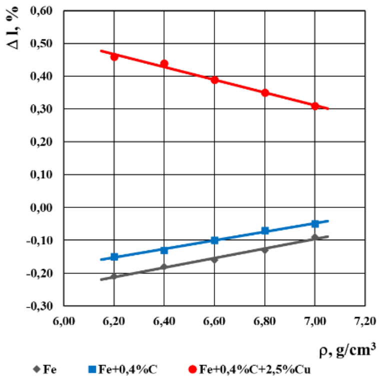

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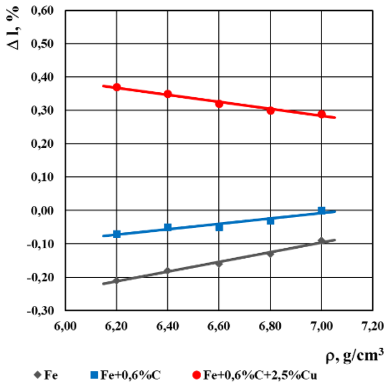

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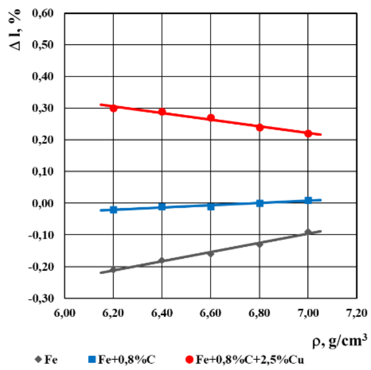

d.

Fig.4. Distribution of the values for the linear changes depending on the density and the carbon concentration of ASC 100.29 iron powder samples a – 0,2%C; b – 0,4%C; c – 0,6%C; d – 0,8%C

Copper is an element that leads to an increase in the size of powder blanks [11], [15], [16]. The copper melted in the sintering process penetrates the boundaries of the iron grains. This copper film replaces the hard boundaries between the grains, resulting in an expansion of the samples. In addition, the penetration of the liquid phase into the polycrystalline system leads to its destruction and its separation into individual grains. This can lead to expansion of the blanks in cases where the torn particles are irregularly shaped and cannot be compacted into the volume that encloses them. The copper in the sintering process dissolves in the iron particles, forming in the matrix an ε-phase (a solid solution of the iron in the copper), which in turn displaces the iron particles and causes an increase in the size of the blanks – fig.5.

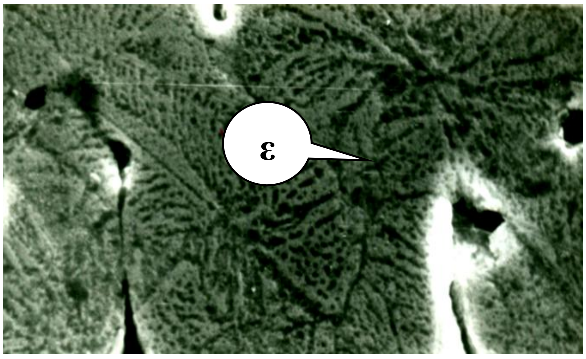

Fig.5. Location of the ε phase along the iron grain boundaries

It is for these reasons that in the samples with a minimum amount of carbon – 0.2%, a significant increase in the size of the blanks is observed, which is not affected by their density. For samples of iron powder NC100.24, this increase is in the range +0.20÷+0.22%, and for those from powder SC 100.26 it is +0.25÷+0.27%. The largest increase was recorded in samples of iron powder ASC 100.29 - +0.40÷+0.50%.

As the carbon concentration increases from 0.2 to 0.8%, a decrease in the size of the blanks is noticed, and those of iron powder NC100.24 undergo a change of +0.06÷+0.12%. In the case of SC 100.26 samples, the change in size growth is in the range of +0.05÷+0.08%, and in the case of ASC 100.29 samples - +0.20%.

This compensating influence of the carbon concentration on the „copper growth" of the powder billets can be attributed to the fact that the carbon in the iron matrix prevents the penetration of the molten copper on the surface of the iron particles and this reduces its solubility in the iron. In addition, carbon is an element that reduces the diffusion coefficient of copper in iron [10,16] and increases the dihedral angle θ between iron grains and liquid copper – fig.6. This leads to an intensification of the sintering process in the presence of a liquid phase.

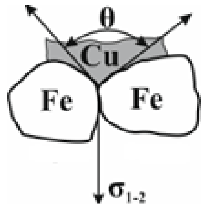

Fig.6. Scheme of the formation of the bilateral angle - θ, of the solid (Fe) / liquid (Cu) phase boundary

All these factors lead to limiting the size growth of copper-alloyed blanks

IV. CONCLUSION

The following more important conclusions can be drawn from the research conducted and the results obtained:

- After sintering of samples of pure iron, the change in linear dimensions has a negative sign, therefore a shrinkage of the blanks is observed, which in samples with a density of 6.20g/cm^3 is of the order of -0.14÷-0.20%;
- As the density of the samples increases from 6.20g/cm^3 to 7.00 g/cm^3, the internal porosity of the blanks decreases, and hence the possibility that the samples undergo a large change in their dimensions during the coalescence of the dust particles.
- The addition of carbon to the matrix leads to a decrease in the compressibility of the blanks, but despite this, at a carbon concentration of 0.2%, the tendency for the change in dimensions to be in the negative part remains.
- In small concentrations - 0.2÷0.6%, carbon leads to a reduction in the dimensions of the samples similar to those of pure iron. At a carbon concentration above 0.6%, however, a change in the linear dimensions of the samples in a positive direction - towards their growth - is found.
- The carbon concentration at which the details of the Fe-C system will not suffer linear changes or they will be insignificant is 0.4÷0.6%.
- During the sintering process, copper dissolves in the iron particles, forming an ε-phase (a solid solution of iron in copper) in the matrix. It displaces the iron particles and causes an increase in the size of the blanks.
- The compensating influence of the carbon concentration on the "copper growth" of the powder billets can be explained by the fact that the carbon in the iron matrix prevents the penetration of the molten copper on the surface of the iron particles and this reduces the solubility in the iron as well.

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