Comparison of magnetic and mechanical properties of powder Fe-Cr-Co alloys with different cobalt content obtained at various sintering temperatures

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Abstract. In present work, we studied the effect of sintering temperature and thermomagnetic treatment (TMT) on the magnetic hysteresis characteristics of the Fe-30Cr-8Co, Fe-30Cr-12Co and Fe-30Cr-16Co (wt. %) powder alloys. All investigated powder alloys, sintered at temperature range of 1100 - 1400 °C have a high level of magnetic properties ensuring their technical application. The highest values of magnetic properties for all three investigated powder alloys are observed after sintering at 1350 °C: \( B_r = 1,28 \) T, \( H_c = 44,3 \) kA/m, \( BH_{(\text{max})} = 39 \) kJ/m\(^3\) - for Fe-30Cr-8Co alloy; \( B_r = 1,28 \) T, \( H_c = 48,2 \) kA/m, \( BH_{(\text{max})} = 39,2 \) kJ/m\(^3\) - for Fe-30Cr-12Co alloy and \( B_r = 1,23 \) T, \( H_c = 54,9 \) kA/m, \( BH_{(\text{max})} = 37,6 \) kJ/m\(^3\) - for Fe-30Cr-16Co alloy. For Fe-30Cr-8Co alloy it was observed that additional annealing at 500 and 480 °C slightly increases the magnetic properties. For Fe-30Cr-12Co and Fe-30Cr-16Co alloys, there is no clear tendency regarding the effect of additional annealing on the magnetic properties. Compression tests of powder alloys Fe-30Cr-8Co and Fe-30Cr-16Co sintered in the temperature range 1100 - 1400 °C have shown that the yield strength \( \sigma_{0.2} \) of the Fe-30Cr-16Co alloy samples (970 – 1080 MPa) are higher compared to the Fe-30Cr-8Co alloy (710 – 930 MPa) throughout the entire sintering temperature range but Fe-30Cr-8Co alloy have a better ductility compared with Fe-30Cr-16Co alloy. Maximum values of \( \sigma_{0.2} \) were obtained on samples sintered at 1100 °C.

1. Introduction
Most of the known classes of hard magnetic materials with high values of magnetic characteristics (for example, alloys based on Nd-Fe-B and Sm-Co systems) are characterized by relatively low mechanical properties that limit their use in a number of technical devices. These materials have low strength and ductility, which also complicates the process of their machining. For this reason, they have limitations on their application, in particular, they cannot be used in units of equipment experiencing high mechanical stress (for example, rotors of hysteresis motors, etc.). Fe-Cr-Co alloys in strength terms clearly stand out in a positive direction, outperforming both based on rare earth and Alnico magnets [1].

In the last decade, interest in Fe-Cr-Co alloys has significantly increased, as evidenced by the increase in the number of scientific publications [2–8]. This is largely due to the development of powder technology for producing these alloys. Modern trends in the production of hard magnetic materials indicate an increase in the percentage of small-scale production, and in this case, the powder metallurgy method has a number of advantages: a high material utilization rate, high precision in molding products and a significant reduction in the volume of mechanical processing. For Nd-Fe-B,
Sm-Co and AlNiCo hard magnetic alloys, powder metallurgy is widely used in production, while for Fe-Cr-Co alloys in the overwhelming majority of cases, melting and casting technology is used. In recent years, various aspects of the synthesis of powder Fe-Cr-Co alloys have been investigated, such as optimization of the temperature-time sintering modes in order to reduce the residual porosity of products and maintain the homogeneity of the chemical composition [9-11], the use of ferroalloy powders (ferro-silicon, ferro-boron, ferro-titanium, etc.) for stimulating sintering due to contact melting and the formation of a liquid phase [12–13]. Several methods of modern powder metallurgy for obtaining Fe-Cr-Co alloys such as the spark plasma sintering method [14] and injection molding of powder compositions (PIM or MIM technologies) [15] were proposed.

Recent studies have shown that Fe-Cr-Co powder alloys with a low and an average cobalt content allows sintering at relatively low temperatures about 1100 – 1200 °C while maintaining high performance values and absence of undesirable phase precipitates [16]. Low-cobalt Fe-Cr-Co alloys are of particular interest, since they have a much lower cost due to decreasing cobalt content in the system.

In present work, we studied the effect of sintering temperature and thermomagnetic treatment (TMT) on the magnetic hysteresis characteristics of the Fe-30Cr-8Co, Fe-30Cr-12Co and Fe-30Cr-16Co (wt. %) powder alloys. Comparison of deformation and yield stress curves after compression tests of Fe-30Cr-8Co and Fe-30Cr-16Co alloy samples obtained at different sintering temperatures was made.

2. Materials and methods

Powder samples of the composition Fe-30Cr-8Co, Fe-30Cr-12Co и Fe-30Cr-16Co were made from industrial high-purity iron powder with a particle size of 10-20 μm, chromium powder with particles less than 50 μm in size and cobalt powder with particles less than 10 μm in size.

The powders were mixed in a C 2.0 turbulent mixer, pressing was performed on a KNUTH HP15 press in a detachable cylindrical matrix with a diameter of 13.6 mm at a pressure of 600 MPa. The height of the compacts was about 20 mm. Sintering was carried out in a shaft furnace in a vacuum of no worse than 10^-2 Pa with a holding time of 2.5 hours at maximum temperature. The density of the samples was determined by hydrostatic weighting. Heating for quenching was carried out in a Nabertherm HT-16/16 high-temperature muffle furnace. Sintered powder samples were quenched from 1300 °C in water. The TMT was carried out in the standard laboratory furnace equipped with armored electromagnet. Magnetic hysteresis properties were measured on a Permagraph L automatic computer controlled measuring instrument. Mechanical compression tests were performed on an Instron 3382 universal testing machine.

3. Results and discussion

Green compacts had a relative density of about 80%. Sintering of the compacts was carried out in the temperature range 1100 - 1400 °C (isothermal holding time - 150 min). After sintering, all three investigated alloys have good relative density (93-95%) even at a low sintering temperature of 1100 °C. With increasing sintering temperature, the relative density values of the alloys increase up to 98 - 98.5% at temperatures of 1350 - 1400 °C.

Heat treatment of the sintered Fe-Cr-Co samples consists of preliminary quenching from 1300 °C and the stage of TMT. As a result of TMT, a high-temperature α-solid solution decomposes into two isomorphic bcc α1 and α2 solid solutions. This leads to a redistribution of components in the system and the formation of a structure that determines the magnetic properties. It is known that the cobalt content in the system affects the kinetics of phase transformations and the possibility of obtaining high magnetic properties, respectively. The kinetics of spinodal decomposition slows down rapidly with decreasing cobalt content. Therefore, for low-cobalt Fe-Cr-Co alloys is often difficult to find the optimal time of TMT. The heat treatment of the alloys studied in this work consisted of a two-stage cooling from a temperature of about 635 - 670 °C down to 580 - 600 °C, depending on the alloy, and subsequent cooling down to 500 °C at rates V1 и V2, respectively (see Table 1). The magnetic field
during TMT was applied along the axis of easy magnetization, coinciding with the axis of the cylindrical sample.

**Table 1.** Heat treatment parameters of Fe-30Cr-8Co, Fe-30Cr-12Co and Fe-30Cr-16Co powder alloys

| Alloy          | Cooling 1            | Cooling 2            | Total TMT time |
|---------------|----------------------|----------------------|----------------|
| Fe-30Cr-8Co   | \( V_1 = 4-8 \, ^\circ C/h \) | \( V_2 = 4-8 \, ^\circ C/h \) | 20-45 h        |
| Fe-30Cr-12Co  | \( V_1 = 18-22 \, ^\circ C/h \) | \( V_2 = 4-8 \, ^\circ C/h \) | 15-25 h        |
| Fe-30Cr-16Co  | \( V_1 = 18-24 \, ^\circ C/h \) | \( V_2 = 6-8 \, ^\circ C/h \) | 13-20 h        |

According to Table 1, the total TMT time of the Fe-30Cr-8Co alloy is significantly higher compared to the other two alloys. The rate of the first cooling in a magnetic field \( V_1 \) for the Fe-30Cr-8Co alloy is almost identical to the second cooling rate \( V_2 \), while for the Fe-30Cr-12Co and Fe-30Cr-16Co alloys \( V_1 \) is several times higher than \( V_2 \). Redistribution of alloy components as a result of spinodal decomposition can occur for a long time. Thereby, after basic heat treatment, the material is often subjected to an additional series of prolonged annealing in the temperature range of 450 - 500 °C. Additional heat treatment provides the necessary redistribution of alloy components between decomposition products in accordance with the equilibrium diagram, which can also lead to an increase of the magnetic properties. For Fe-30Cr-8Co alloy it was observed that additional annealing at 500 and 480 °C slightly increases the magnetic properties (see Figure 1).

![Figure 1. Dependence of \( H_c \) and \( (BH)_{\text{max}} \) of Fe-30Cr-8Co alloy samples sintered at different temperatures on the TMT time.](image-url)
The values of the coercive force \( H_c \) and the maximum energy product \((BH)_{\text{max}}\) of the Fe-30Cr-8Co alloy samples sintered at different temperatures (Figure 1) slightly increase with increasing heat treatment time. The residual induction \( B_r \) remains approximately at the same level. For Fe-30Cr-12Co and Fe-30Cr-16Co alloys, there is no clear tendency regarding the effect of additional annealing on the magnetic properties. Thus, the obtained experimental results are in agreement with the known data on the kinetics of spinodal decomposition in the system and the effect of the cobalt content on the total heat treatment time [17]. Low-cobalt Fe-Cr-Co alloys require a longer heat treatment, which lowering their processability. However, the magnetic properties of the Fe-30Cr-8Co alloy after basic heat treatment for about 30–40 hours already have a level sufficient for technical use. For comparison, the magnetic properties of cast alloy 28H10KA according to GOST 24897-81: \( B_r = 1.1 \) T, \( H_c = 38 \) kA / m, \((BH)_{\text{max}} = 26 \) kJ / m³.

The maximum values of the magnetic properties of Fe-30Cr-8Co, Fe-30Cr-12Co and Fe-30Cr-16Co alloys sintered in the temperature range 1100 - 1400 °C are presented in Table 2. Individual hysteresis loops of the studied alloys samples are shown in Figure 2.

**Table 2.** The maximum values of the magnetic properties of Fe-30Cr-8Co, Fe-30Cr-12Co and Fe-30Cr-16Co powder alloys, obtained at different sintering temperatures

| Alloy       | Sintering temperature, °C | \( B_r \), T | \( H_c \), kA/m | \((BH)_{\text{max}}\), kJ/m³ |
|-------------|---------------------------|-------------|---------------|-----------------------------|
| Fe-30Cr-8Co |                           | 1,19        | 43,9          | 30,4                        |
| Fe-30Cr-12Co| 1100                      | 1,19        | 46,8          | 32,4                        |
| Fe-30Cr-16Co|                           | 1,19        | 48,6          | 32,6                        |
| Fe-30Cr-8Co |                           | 1,25        | 42,7          | 35,2                        |
| Fe-30Cr-12Co| 1250                      | 1,24        | 45,9          | 33,8                        |
| Fe-30Cr-16Co|                           | 1,21        | 51,8          | 33,3                        |
| Fe-30Cr-8Co |                           | 1,28        | 44,3          | 39,0                        |
| Fe-30Cr-12Co| 1350                      | 1,28        | 48,2          | 39,2                        |
| Fe-30Cr-16Co|                           | 1,23        | 54,9          | 37,6                        |
| Fe-30Cr-8Co |                           | 1,28        | 43,6          | 38,0                        |
| Fe-30Cr-12Co| 1400                      | 1,26        | 47,4          | 37,5                        |
| Fe-30Cr-16Co|                           | 1,21        | 54,7          | 35,7                        |
From the analysis of the obtained results, it follows that the highest values of magnetic properties for all three investigated powder alloys are observed after sintering at 1350 °C. A slight decrease in the level of magnetic properties in the samples sintered at 1400 °C is most likely explained by the partial chromium evaporation and the chemical composition change in the near-surface layer. An increase in the residual porosity after sintering at 1250 and 1100 °C monotonically decreases the residual induction $B_r$ of all three alloys. The coercive force $H_c$ is less susceptible to changes as residual porosity of the samples increases. Moreover, the coercive force primarily depends on the degree of homogeneity of the powder composition. As can be seen from Table 2, the maximum values of the coercive force of the Fe-30Cr-16Co alloy monotonically decrease at the lower sintering temperatures, similarly to the dependence of the residual induction, while the values of the coercive force of the Fe-30Cr-8Co and Fe-30Cr-12Co alloys differ slightly from those for samples sintered at 1350 °C. The dependence of the maximum energy product $(BH)_{max}$ on the sintering temperature is similar to the dependence of the residual induction.

Compression tests were carried out on samples of powder alloys Fe-30Cr-8Co and Fe-30Cr-16Co sintered in the temperature range 1100 - 1400 °C. Deformation curves of Fe-30Cr-8Co and Fe-30Cr-16Co alloys samples are presented at Figure 1.
Figure 3. Deformation curves during compression tests of Fe-30Cr-8Co (a, c) and Fe-30Cr-16Co (b, d) alloys samples sintered at temperatures of 1100 °C (a, b) and 1400 °C (c, d) after a full cycle of heat treatment.

All test samples were deformed to the ultimate load of the testing machine. No fracture was observed on the Fe-30Cr-8Co alloy samples (Figure 3a, c). On the Fe-30Cr-16Co alloy samples (Figure 3b, d), cracks appeared at a stress of about 2100 - 2200 MPa. Cobalt has an embrittling effect in the Fe-Cr-Co system, so lowering cobalt content improves ductility. A similar character of the deformation curves was observed for the investigated alloys samples, sintered at other temperatures. The sintering temperature dependence of the yield strength $\sigma_{0.2}$ is shown in Figure 4.

Yield strength values of the Fe-30Cr-16Co alloy samples (970 – 1080 MPa) are higher compared to the Fe-30Cr-8Co alloy (710 – 930 MPa) in the entire investigated range of sintering temperatures (Figure 4). In addition, it should be noted that, with an increase in the sintering temperature, the values of $\sigma_{0.2}$ for both alloys decrease. This is most likely due to the coarsening of the grain with an increase in the sintering temperature.

4. Conclusion
The magnetic properties of the Fe-30Cr-8Co, Fe-30Cr-12Co and Fe-30Cr-16Co powder alloys, sintered at temperatures of 1100 - 1400 °C have a high level ensuring their technical application.
Properties exceed the characteristics of analogues obtained by casting technology. The highest values of magnetic properties for all three investigated powder alloys are observed after sintering at $1350 \, ^\circ\text{C}$.

Low-cobalt Fe-Cr-Co alloys have a lower cost but require a longer heat treatment, which lowering their processability.

For Fe-30Cr-8Co alloy it was observed that increasing heat treatment time due to additional annealing at temperature range of $450 – 500 \, ^\circ\text{C}$ slightly increases the magnetic properties. For Fe-30Cr-12Co and Fe-30Cr-16Co alloys, there is no clear tendency regarding the effect of additional annealing on the magnetic properties. From the point of view of rationality, in most cases, the above annealing is not required for the investigated alloys.

No fracture was observed on the Fe-30Cr-8Co alloy samples after full heat treatment cycle during compression tests. On the Fe-30Cr-16Co alloy samples cracks appeared at a stress of about $2100 - 2200 \, \text{MPa}$. Low-cobalt Fe-Cr-Co alloys have a better ductility compared with Fe-Cr-Co alloys with average cobalt content.

Yield strength $\sigma_{0,2}$ of the Fe-30Cr-16Co alloy samples ($970 – 1080 \, \text{MPa}$) are higher compared to the Fe-30Cr-8Co alloy ($710 – 930 \, \text{MPa}$) in the entire investigated range of sintering temperatures. In addition, it should be noted that, with an increase in the sintering temperature, the values of $\sigma_{0,2}$ for both alloys decrease. This is most likely due to the coarsening of the grain with an increase in the sintering temperature.

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