Effect of sintering temperature on magnetic hysteresis characteristics of powder alloy Fe-30Cr-8Co (wt.%)

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Abstract. In this work, we studied the effect of sintering temperature (1100 - 1400 °C) and thermomagnetic treatment (TMT) on the magnetic hysteresis characteristics of the Fe-30Cr-8Co alloy, obtained by powder technology. It has been shown that at sintering temperatures of 1200 °C and below, it is possible to obtain materials with high magnetic characteristics that ensure their technical application. Properties exceed the characteristics of analogues obtained by casting technology. In the range 1200 - 1250 °C, the largest increase in the density of samples is observed with an increase in sintering temperature with a simultaneous increase in the values of magnetic characteristics. It was revealed that the total duration of heat treatment for 40-80 hours is sufficient for the formation of the necessary structure that determines the magnetic properties of the material. Long-term heat treatment (up to 160 hours) contributes to a higher level of magnetic properties.

1. Introduction
Hard magnetic materials are widely used as permanent magnets in various fields of technology, including power electronics (magnetrons, klystrons, travelling-wave lamps, etc.), electrical engineering (DC motors), instrumentation (magnetoelectric devices), and mechanical engineering (various types of separators), radio equipment (microphones and audio speakers), communications, medicine and. Etc [1-2]. Most classes of hard magnetic materials (cast alloys based on the Fe-Ni-Al-Co-Cu-Ti system, alloys based on the rare-earth compounds SmCo5, Sm2Co17, Nd2Fe14B, magnetically hard barium and strontium ferrites) are characterized by relatively low mechanical (strength and plastic properties). However, in some cases of important technical application, magnetically hard material not only requires high magnetic hysteresis properties (residual induction $B_r$, coercive force $H_c$, maximum energy product $(BH)_{max}$) but also high strength ($\sigma_n \sim 600-800$ MPa).

Magnetic alloys of the Fe-Cr-Co system occupy a deserved place among hard magnetic materials in connection with their high mechanical properties in the presence of a sufficiently high level of magnetic hysteresis properties. The production of Fe-Cr-Co hard alloys is carried out in two ways - by casting and by powder metallurgy. The powder method has a number of additional advantages compared to casting technology: the ability to precisely control the chemical composition of alloys, an increased metal utilization rate in production, high productivity with large production volumes, and the possibility of economical manufacturing of piece lots of simple-shaped magnets. In this case, the magnetic hysteresis characteristics of powder magnets are not inferior to those of cast magnets [3-4].

The high vacuum sintering temperatures used in the production of magnetic alloys of the Fe-Cr-Co system cause evaporation of elemental components (mainly chromium) from the surface of the workpieces, which leads to a inhomogeneity of the chemical composition and deterioration of the material characteristics. Recent studies of powder Fe-Cr-Co alloys have shown that alloys with a low
cobalt content can be sintered at relatively low temperatures while maintaining high performance values [5].

In this work, we studied the effect of sintering temperature and thermomagnetic treatment on the magnetic hysteresis characteristics of the Fe-30Cr-8Co powder alloy. It has been shown that at sintering temperatures of 1200 °C and lower it is possible to obtain materials with high magnetic characteristics.

2. Materials and methods
Powder samples of the composition Fe-30Cr-8Co 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 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 weighing. Quenching was carried out in a Nabertherm HT-16/16 high-temperature muffle furnace. Sintered powder samples were quenched from 1300 °C in water. Heat treatment was carried out in a muffle furnace and magnetic hysteresis properties were measured on a Permagraph L. hysteresograph. Photographs of the microstructure were obtained with an Olympus GX51 optical microscope. X-ray phase analysis was performed on a SHIMADZU XRD-6000 vertical x-ray diffractometer with monochromatized copper radiation.

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 (sintering time - 150 min). The dependence of the relative density of sintered samples on sintering temperature is shown in Figure 1.

![Figure 1](image)

Figure 1. Dependence of the relative density of Fe-30Cr-8Co powder alloy samples on the sintering temperature.

Figure 1 shows that the alloy has a sufficiently high relative density (94.5%) even at low sintering temperatures of the order of 1100 - 1150 °C. The increase in density can be observed in the temperature range 1200 - 1250 °C, when the density reaches 98%. Heat treatment of the Fe-30Cr-8Co alloy samples, sintered at various temperatures, was carried out and magnetic properties were measured. The magnetic field during TMT was applied along the axis of easy magnetization, coinciding with the axis of the cylindrical sample. The results of measurement of the magnetic properties of the Fe-30Cr-8Co alloy samples, sintered at various temperatures are presented in table 1 and some hysteresis loops in Figure 2.
Table 1. Magnetic hysteresis properties of the Fe-30Cr-8Co alloy.

| Alloy   | Sintering temperature, °C | $B_r$, T | $H_c$, kA/m | $(BH)_{max}$, kJ/m$^3$ |
|---------|---------------------------|----------|-------------|------------------------|
| Fe-30Cr-8Co | 1100                      | 1,18 – 1,19 | 40,7 – 43,4 | 27,6 – 30,2             |
|         | 1150                      | 1,18 – 1,19 | 41,5 – 42,8 | 29,1 – 31,0             |
|         | 1200                      | 1,18 – 1,21 | 40,0 – 41,9 | 31,8 – 33,0             |
|         | 1250                      | 1,21 – 1,25 | 40,0 – 42,7 | 32,9 – 35,2             |
|         | 1300                      | 1,22 – 1,26 | 40,3 – 42,4 | 34,3 – 36,2             |
|         | 1400                      | 1,26 – 1,28 | 40,8 – 43,6 | 36,1 – 38,0             |

Figure 2. Hysteresis loops of Fe-30Cr-8Co samples, sintered at a temperature of 1100 (a), 1200 (b), 1300 (c) and 1400 °C (d) after a complete TMT cycle.

From the analysis of these results it follows that the magnetic properties of the powder alloy under all sintering conditions are at a high level, ensuring their technical application and exceed the characteristics of analogues obtained by casting technology. For example, the magnetic properties of alloy 28H10KA according to GOST 24897-81: $B_r = 1.1$ T, $H_c = 38$ kA / m, $(BH)_{max} = 26$ kJ / m$^3$. It should also be noted that there is an increase in the magnetic characteristics in the temperature range of 1200 - 1250 °C. This fact is largely explained by an increase in the density of samples in the same temperature range. A further increase in sintering temperature up to 1400 °C gives a small increase in the residual induction $B_r$ and maximum energy product $(BH)_{max}$ while the coercive force $H_c$ remain at the same level.

It should be noted that the Fe-30Cr-8Co powder alloy has a high level of magnetic properties at low sintering temperatures of 1100 - 1150 °C. X-ray phase analysis of the samples sintered at a temperature of 1100 °C (Figure 3) shows the presence of only the α phase after a complete TMT cycle. A comparison of magnetic properties with the data of our previous studies about phase composition [5] shows that the absence of undesirable phases during sintering is an important factor for the formation of a high level of magnetic properties in Fe-Cr-Co powder alloys. In addition, sintering in
the field of formation of a single-phase bcc $\alpha$-solid solution also reduces the residual porosity in samples.

![Figure 3](image3.png)  
**Figure 3.** X-ray diffraction pattern of Fe-30Cr-8Co powder alloy sample, sintered at a temperature of 1100 °C after a complete TMT cycle.

![Figure 4](image4.png)  
**Figure 4.** Photograph of the microstructure of Fe-30Cr-8Co powder alloy sample, sintered at a temperature of 1100 °C after a complete TMT cycle.

Microstructure of the Fe-30Cr-8Co powder alloy sample, sintered at a temperature of 1100 °C, is presented in Figure 4. A mixture of aqua regia and glycerin was used as an etchant. Aqua regia is the main, recommended etchant for alloys of this system, however, when it is used without glycerin, structure blur often occurs. As experimental studies have shown, glycerin slows down the action of aqua regia and allows better identification of the microstructure. The microstructure of the Fe–30Cr–8Co powder alloy (Figure 4) after the complete TMT cycle consists mainly of grains with a diameter of 20–40 μm; dark areas in the photographs are identified as pores. The formation of the structure, responsible for the magnetic properties, occurs as a result of a certain heat treatment of the sintered sample. Heat treatment modes are presented in Table 2.

**Table 2.** Heat treatment modes of a Fe-30Cr-8Co powder alloy, sintered at a temperature of 1400 °C.

| Mode | Stage 1                                      | Stage 2                      | Stage 3                      | Stage 4                      |
|------|---------------------------------------------|------------------------------|------------------------------|------------------------------|
| 1    | 670°C(10 min) + cooling to 500°C with $V= 4 \degree C/h$ | -                            | -                            | -                            |
| 2    | 670°C(10 min) + cooling to 500°C with $V= 4 \degree C/h$ + annealing at 500°C(20 hours) | -                            | -                            | -                            |
| 3    | 670°C(10 min) + cooling to 500°C with $V= 4 \degree C/h$ + annealing at 500°C(20 hours) + cooling to 480°C(20 hours) | -                            | -                            | -                            |
| 4    | 670°C(10 min) + cooling to 500°C with $V= 4 \degree C/h$ + annealing at 500°C(20 hours) + cooling to 480°C(20 hours) + 460°C(20 hours) | -                            | -                            | -                            |
| 5    | 670°C(10 min) + cooling to 500°C with $V= 4 \degree C/h$ + annealing at 500°C(20 hours) + cooling to 480°C(20 hours) + 480°C(40 hours) | -                            | -                            | -                            |
| 6    | 670°C(10 min) + cooling to 500°C with $V= 4 \degree C/h$ + annealing at 500°C(20 hours) + cooling to 480°C(20 hours) + 480°C(80 hours) | -                            | -                            | -                            |
Heat treatment 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 \( \alpha_1 \) and \( \alpha_2 \) solid solutions [6]. This leads to the redistribution of components in the system and the formation of a structure that determines the magnetic properties. Heat treatment of the Fe-30Cr-8Co alloy samples, sintered at a temperature of 1400 °C, was carried out according to six modes (table 2) and magnetic properties were measured. Total TMT time increased from 40 hours (mode 1) to 160 hours (mode 6).

The dependences of \( H_c \) and \((BH)_{\text{max}}\) on the heat treatment time are presented in Figure 5. The residual induction \( B_r \) is independent of the TMT time and has a value of about 1.27 T.

![Figure 5](image)

**Figure 5.** Dependence of \( H_c \) (a) and \((BH)_{\text{max}}\) (b) of Fe-30Cr-8Co alloys samples, sintered at a temperature of 1400 °C from the TMT time.

The coercive force \( H_c \) of the alloy (Figure 5a) grows with increasing heat treatment time. The dependence of the maximum energy product \((BH)_{\text{max}}\) on the heat treatment time (Figure 5b) has a similar form to the curve for the coercive force.

Samples subjected to TMT for 40-80 hours have the necessary level of magnetic hysteresis properties for technical use. However, if the consumer needs a higher level of magnetic properties of the products, then a long heat treatment (up to 160 hours) contributes to its production. From an economic point of view, heat treatment of alloys lasting less than 100 hours is sufficient.

4. Conclusion
The magnetic properties of the Fe-30Cr-8Co powder alloy, sintered at temperatures of 1100 - 1400 °C are at a high level ensuring their technical application. Properties exceed the characteristics of analogues obtained by casting technology.

In the temperature range 1200-1250 °C, the largest increase in the density of samples is observed with an increase in sintering temperature with a simultaneous increase in the values of magnetic characteristics.

TMT for 40-80 hours provides a sufficient level of magnetic hysteresis properties. Long-term heat treatment (up to 160 hours) contributes to a higher level of magnetic properties.

Acknowledgements
This study was supported by the Russian Foundation for Basic Research (project no. 18-03-00666).

References
[1] Artamonov E V, Libman M A, and Rudanovskii N N 2007 Magnetically Hard Materials for the Motors of Synchronous Hysteresis Electric Motors *Steel in Transl.* 37 547-51
[2] S Jin, G Y Chin and Wonsiewicz B C 1980 A Low Cobalt Ternary Cr-Co-Fe Alloy for Telephone Receiver Magnet Use *IEEE Trans. on Magn.* 16 3187-92
[3] Alymov M I, Ankudinov A B, Zelenskii V A, Milyaev I M, Yusupov V S and Vompe T A 2014 The effect of surfactant admixture during milling on pressing, sintering, and magnetic properties of FeCrCoMoW alloy Inorg. Mat. Appl. Res. 5 530-5

[4] Shatsov A A, Ryaposov I V and Kozvonin V A 2017 Concertation-Inhomogeneous Hard Magnetic Alloys of the Fe – Cr – Co System with Elevated Content of Cobalt and Boron Met. Sci. and Heat Treat. 59 45-9

[5] Ustyukhin A S, Ankudinov A B, Zelenskii V A, Milyaev I M, Ashmarin A A and Alymov M I 2018 Phase composition of powder hard magnetic Fe–30Cr–(8–24)Co alloys sintered under different temperature conditions Dokl. Phys. Chem. 482 140-4

[6] Kaneko H, Homma M and Nakamura K 1971 New Ductile Permanent Magnet of Fe-Cr-Co System AIP Conf. Proceedings. Magn. Magn. Mater. 1088–92