Effect of mixing duration and sintering temperature on the magnetic properties of hard magnetic powder alloy Fe-30Cr-8Co (wt.%) 

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Abstract. Effect of mixing duration and sintering temperature on the magnetic properties of hard magnetic powder alloy Fe-30Cr-8Co (wt.%) was studied. The obtained magnetic hysteresis properties of the hard magnetic powder alloy are better than as for industrial Fe-Cr-Co alloy which cobalt content is twice bigger. The optimum sintering temperature was 1350-1400 °C. At a sintering temperature of 1400 °C for 2.5 hours the mixing duration does not affect on the magnetic hysteresis properties. This technological operation is not critical.

1. Introduction

Hard magnetic materials of Fe-Cr-Co system have a special place among the hard magnetic materials due to their high mechanical (strength and plastic) characteristics along with a sufficiently high level of magnetic hysteresis properties (residual induction $B_r$, coercive force $H_c$, maximum energy product $(BH)_{max}$) [1].

Currently, special attention of researchers is attracted to low-cobalt (7-10 wt. % Co) hard magnetic Fe-Cr-Co alloys [2, 3] due to the fact that price of the cobalt in the global markets has been dramatically rise.

The previous investigations of the powder low cobalt hard magnetic Fe-Cr-Co alloys [4], that were held in the decade of 1980s, showed the fundamental possibility of obtaining very high magnetic hysteresis properties, nevertheless they have not received commercial application up to now.

The aim of the present work was to determine the effect of mixing duration and sintering temperature on the magnetic properties of hard magnetic powder alloy Fe-30Cr-8Co (wt.%).

Our findings open pathways towards fine production of Fe-Cr-Co hard magnetic alloys with controlled characteristics for distinctive applications.

2. Materials and methods

2.1. Materials

The samples of hard magnetic Fe-30Cr-8Co (wt. %) powder alloy were prepared by the methods of powder metallurgy. Powders of iron (VS), chrome (PHS-1), cobalt (PK-1N) of commercial purity have been used.
2.2. Preparation of the Samples

The powder particles were mixed in turbulence mixer C 2.0 “Turbula” in the interval 1-60 minutes. The powder was compacted by single-ended dry pressing at 600 MPa in a hardened steel die on a manual press KNUTH-130042. The pressing die had internal diameter 13.6 mm.

Then samples have been sintered for 2.5 hours at different temperatures in a shaft furnace SShV-1,25/24-I1 in 10⁻² Pa vacuum. The height of the cylinder samples was 20 mm.

2.3. Characteristics of the samples

The sintered density of the hard magnetic Fe-30Cr-8Co (wt. %) powder alloy samples was determined by hydrostatic weighing. Then it has been correlated with the theoretical density of the alloy.

The treatment of samples was carried out in the standard laboratory furnace equipped with armored electromagnet. The magnetic field strength was 4000 Oe. The temperature was kept and monitored by proportional–integral–derivative controller. Heat treatment was carried out in standard laboratory muffle furnace equipped with the controller PROTAR 101.

Magnetic hysteresis properties including residual induction Bᵢ, coercive force Hᵢ and maximum energy product (BH)ₘₐₓ for the solution-treated alloys were determined from B-H loops automatically recorded by the Permagraph L EP-3 hysteresis graph. The measurement error for coercive force Hᵢ and residual induction Bᵢ was 3 %, and it was 6 % for maximum energy product (BH)ₘₐₓ.

The scheme of the heat treatment is shown in the figure 1.

![Figure 1](image1.png)

**Figure 1.** Schematic diagram of the heat treatment of the Fe-30Cr-8Co alloy.

The samples were quenched at 1100 °C in water and annealed from 670 °C till 580 °C at the rate of 4 °C/h in a 4000 Oe magnetic field. Further controlled cooling was conducted up to 500 °C without magnetic field at the rate of 4 °C/h. There was also additional annealing till 460 °C.

The x-ray diffraction studies were performed using a DRON-3M diffractometer, FeKᵢ radiation.

3. Results and discussion

3.1. Effect of Sintering Temperature

Density of samples of Fe-30Cr-8Co powder alloys and magnetic hysteresis properties of Fe-30Cr-8Co alloys versus sintering temperature in the range 1100 – 1400 °C are shown in figures 1 and 2, respectively.

One can see that there is a direct correlation between the increasement of the density of sintered samples with sintering temperature increasement and magnetic hysteresis properties increasement.
Relative increment of the residual induction $B_r$ and coercive force $H_c$ at sintering temperature at 1400 °C as compared to those obtained for 1100 °C are practically equal to the relative increment of density. All values increase by about 2%. Moreover, the curve of the temperature dependence of the coercive force has the same appearance as the curve for density change. These facts indicate that the structure of the investigated alloy as a functional magnetic material has been formed after sintering already at 1100 °C. Further sintering temperature increase affects only on the shape of the demagnetization curve. The evidence of this phenomenon is the temperature dependence of the maximum energy product, when the growth $(BH)_{max}$ is disproportionately high in comparison with the increase in density up to 1300 °C.

3.2. The Effect of Mixing Duration
Magnetic hysteresis properties of Fe-30Cr-8Co alloys versus mixing duration after sintering at 1200 °C during 2.5 hours is shown in the figure 4.

The figure 4 shows that the effect of the mixing duration on the magnetic hysteresis properties can be described by logarithmic function. Mixing for more than 15 minutes has practically no significant effect on the magnetic properties.

The x-ray diffraction studies of the samples, sintered in the entire studied range of sintering temperature, revealed that the obtained materials are single-phase materials with a ferrite structure, which is typical for low-cobalt alloys even at a low synthesis temperature. The trace of $\gamma$-phase was not detected. The X-ray diffraction pattern of the sample that was sintered at 1150 °C is typical (figure 5), similar x-ray patterns were obtained at other temperatures up to 1400 °C.

Figure 2. Density of samples of Fe-30Cr-8Co powder alloys versus sintering temperature
Figure 3. Magnetic hysteresis properties of Fe-30Cr-8Co alloys versus sintering temperature: (a) $B_r$, (b) $H_{cb}$ and (c) $(BH)_{max}$.

Figure 4. Magnetic hysteresis properties of Fe-30Cr-8Co alloys versus mixing duration of powder after sintering at 1200 °C: (a) $B_r$, (b) $H_{cb}$ and (c) $(BH)_{max}$. 
Magnetic hysteresis properties of Fe-30Cr-8Co alloys depending on the mixing duration after sintering at 1400 °C are shown in the table 1.

| Time of mixing, min | $B_r, T$ | $H_{dc}, kAm^{-1}$ | $(BH)_{max}, kJm^{-3}$ |
|--------------------|----------|-------------------|----------------------|
| 1                  | 1.245    | 43.5              | 38.2                 |
| 5                  | 1.247    | 43.2              | 37.9                 |
| 15                 | 1.245    | 43.4              | 37.8                 |
| 30                 | 1.248    | 43.4              | 38.1                 |
| 60                 | 1.252    | 43.5              | 38.4                 |

The mixing duration from 1 minute to 60 minutes does not affect on the magnetic properties after sintering at 1400 °C for 2.5 hours.

For samples sintered at 1200 °C and 1400 °C the difference in the behavior of the magnetic characteristics depending on mixing duration is quite explainable. With an increase in the sintering temperature increases by 200 °C, diffusion processes are activated significantly. In particular, the diffusion length increases significantly at a fixed sintering time, which determines the degree of completeness of the sintering process in terms of obtaining a homogeneous alloy. At a temperature of 1200 °C, after sintering samples with a short mixing time, the sintered material is not homogeneous. As a result, magnetic properties have smaller values. At this temperature, the sintering time of 2.5 hours is clearly not enough to create a homogeneous structure of the sintered material from insufficiently mixed powders with a short mixing time. Thus, it is possible to eliminate the heterogeneity in the sintered material and to obtain higher values of magnetic properties only after prolonged mixing.

4. Conclusion
The obtained magnetic hysteresis properties of the hard magnetic Fe-30Cr-8Co (wt. %) powder alloy are better than as for industrial Fe-25Cr-15Co-Al-Nb-V alloy which cobalt content is twice bigger.
The structure of the investigated alloy as a functional magnetic material turns out to be formed at 1100 °C temperature sintering. The resulting materials are single phase materials with a ferrite structure.

The optimum sintering temperature of the powder alloy Fe-30Cr-8Co should be recognized as 1350-1400 °C.

At a sintering temperature of 1400 °C for 2.5 hours the duration time does not affect on the magnetic hysteresis properties of the Fe-30Cr-8Co alloy. Thus, this technological operation is not critical.

Our findings can be used in synthesizing and achieving desired magnetic properties of the hard magnetic Fe-30Cr-8Co powder alloys.

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