Influence of heat treatment on the magnetic properties of Fe-28%Cr-7%Co-2%Mo-0.5%Si powder hard magnetic alloy

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Abstract. The hard magnetic alloy Fe-28%Cr-7%Co-2%Mo-0.5%Si (in wt. %) was prepared by the methods of powder metallurgy. The maximum magnetic properties on the alloy was received after the TMT temperature of 540 °C and was the following: residual induction $B_r$ up to 1.3 T, coercive force $H_c$ up to 40 kAm$^{-1}$ and maximum energy product $(BH)_{max}$ up to 31 kJm$^{-3}$. The hardness of the alloy increases from 228 to 250 HV 30 in the temperature range of spinodal decomposition at 600 – 660 °C. The coefficient of thermal expansion of the alloy is $13.1 \times 10^{-6}$ K$^{-1}$. It was found that the additional annealing of the alloy till 460 °C can increase the coercive force approximately for 6 kAm$^{-1}$.

1. Introduction
Permanent magnets based on the Fe-Cr-Co system are technologically important materials for electric power generation, transportation, power electronics and information technology [1]. They are widely used to manufacture permanent magnets due to their unique combination of magnetic and mechanical properties. Fe-Cr-Co magnetic materials have several advantages to be used as rotor ring for hysteresis motors [2]. The final cost of such magnets depends on amount of cobalt.

Fe-Cr-Co permanent magnet alloys achieve their properties after heat treatment due to decompose by a spinodal decomposition [3] of high temperature BCC $\alpha$ – solid solution into two isomorphic BCC $\alpha_1$ and $\alpha_2$ solid solutions. Besides, high magnetic $\alpha_1$ phase is enriched by Fe and Co and weakly magnetic $\alpha_2$ phase is enriched by Cr [4]. The significant changes in the microstructure and magnetic properties of Fe-Cr-Co system alloys occur during thermomagnetic treatment (TMT) [5]. Magnetic aging greatly improve the magnetic properties [6].

The production of the Fe-Cr-Co system for permanent magnets by the methods of powder metallurgy has advantages over the casting technology [7]. For example, the magnetic properties of the alloy can be increased due to the better compositional control offered by powder metallurgy processing as compared to melt practice [8].

Low cobalt Fe-Cr-Co alloys have better ductility and slower kinetics of phase transformations with comparisons with alloys with 15 % of cobalt and more. As cobalt content is reduced, the kinetics of spinodal decomposition slows down rapidly [9]. The spinodal temperatures of the low cobalt Fe-Cr-Co alloys are found to be higher than the Curie temperatures [10]. The important task is to investigate new low cobalt Fe-Cr-Co alloys for further commercial development.

In the present paper, we have investigated the influence of heat treatment on the magnetic properties of Fe-28%Cr-7%Co-2%Mo-0.5%Si powder hard magnetic alloy.
2. Materials and methods

2.1. Materials
The alloy Fe-28%Cr-7%Co-2%Mo-0.5%Si (in wt. %) was prepared by the methods of powder metallurgy. The commercial purity powders of iron, chrome, cobalt, molybdenum and silicon have been used.

2.2. Preparation of the samples
Particles of the powders were mixed in C 2.0 “Turbula” mixer (Russia). Single-ended dry pressing at 600 MPa in a zinc stearate lubricated hardened steel die on a manual press KNUTH-130042 (Germany) was used to compact the samples. The internal diameter of pressing die was 13.6 mm.

The density of the samples was determined by hydrostatic weighing. The density of raw samples was 78.8 – 79.1 %. Then compacted samples were sintered for 2.5 hours at the temperature of 1420 °C in a shaft furnace SshV-1,25/24-I (Russia) in 10⁻² Pa vacuum. Finally, the sintered density of prepared samples was 98.6 – 99.1 %.

2.3. Characteristics of the samples
The samples of Ø12×20 mm in size were solution treated at the temperature of 1100 °C for 15 minutes, quenched in water, followed by the TMT (H = 4 kOe) and multiple step aging for various times. The TMT was carried out in the standard laboratory furnace equipped with armored electromagnet. The temperature was kept and monitored by proportional–integral–derivative controller. Standard laboratory muffle furnace equipped with the controller PROTAR 101 was used for heat treatment.

Magnetic hysteresis properties (residual induction $B_r$, coercive force $H_c$ and maximum energy product $(BH)_{max}$) were measured by the Permagraph L EP-3 (Germany). The measurement error for coercive force $H_c$ and residual induction $B_r$ was 3 %, and it was 6 % for maximum energy product $(BH)_{max}$. The surface of sample was cleaned from slag using abrasive disk before measurements of the magnetic properties.

The samples were quenched at $T_q = 1100$ °C in water. The heat treatment was carried out by a two-stage continuous cooling starting from above the $\alpha_1+\alpha_2$ miscibility gap as shown schematically in figure 1. The initial cooling rates were equal 5 °C/h from $T_2 = 580$ °C and 3.4 °C/h from 540 °C. The second cooling rates were 2.3 °C/h and 3.6 °C/h from $T_3 = 500$ °C. After aging, there was also additional annealing till 460 °C to increase the magnetic properties. After that all the samples were air cooled from 460 °C to room temperature.

The dilatometric measurements were performed using a dilatometer NETZCH DIL 402 C72G (Germany). The samples were heated to the temperature 1300 °C and cooled to room temperature at the rate of 10 °C/sec with constant argon blow at the rate of 75 ml/min. The coefficient of thermal expansion (CTE) of the Fe-28%Cr-7%Co-2%Mo-0.5%Si alloy was also determined.
3. Results and discussion

3.1. Thermal expansion behavior of the alloy

Figure 2 shows the thermal expansion curve during heating and cooling of the Fe-28%Cr-7%Co-2%Mo-0.5%Si alloy at temperatures between 500 and 700 °C.

According to the phase diagrams and experimental data the phase transformation of $\alpha \rightarrow \alpha_1 + \alpha_2$ would be expected at the temperature range 620 – 640 °C. The TMT parameters were selected based on this temperature.

The average CTE of the alloy in the temperature between 20 °C and 900 °C is $13,1 \times 10^{-6} \text{ K}^{-1}$.

3.2. Hardness of the alloy after spinodal composition

The effect of 30 min annealing in the temperature range of 550 – 1300 °C on hardness of the alloy is shown at the figure 3. The hardness increases from 228 to 250 HV 30 in the temperature range of spinodal decomposition at 600-660 °C. In the temperatures from 900 °C to 1100 °C the hardness is approximately constant and after annealing at 660°C it is similar like after quenching.

3.3. Magnetic properties and additional annealing

Experimental data in table 1 shows magnetic properties that were received after different heat treatment regimes.
Table 1. Magnetic properties of the Fe-28%Cr-7%Co-2%Mo-0.5%Si alloy.

| TMT          | Heat treatment regimes | $B_r$, T   | $H_c$, kAm$^{-1}$ | $(BH)_{max}$, kJm$^{-3}$ |
|--------------|------------------------|------------|-------------------|--------------------------|
| A            | 660 → 580 °C 5 °C/h    | 580 → 500 °C 3.6 °C/h Field (4kOe) No field | 1.22 | 28.4 | 18.6 |
| B            | 660 → 540 °C 3.4 °C/h  | 540 → 500 °C 2.3 °C/h Field (4kOe) No field | 1.24 | 34.0 | 26.9 |

The changing of the temperature at the first step to 540 °C and decreasing of the rate of the cooling to 3.4 °C/h leads to the increase of the magnetic properties of the alloy.

It was found that the additional annealing after 500 °C till 460 °C can increase the magnetic properties of the alloy. The temperature influence on the coercive force $H_c$ of the Fe-28%Cr-7%Co-2%Mo-0.5%Si alloy after 20 hours annealing is shown at the figure 4 and 5.

![Figure 4. Temperature influence on the coercive force of the Fe-28%Cr-7%Co-2%Mo-0.5%Si alloy after A heat treatment and annealing.](image1)

![Figure 5. Temperature influence on the coercive force of the Fe-28%Cr-7%Co-2%Mo-0.5%Si alloy after B heat treatment and annealing.](image2)

Due to slower kinetics of phase transformations in low cobalt Fe-Cr-Co alloy the additional annealing was used to increase the value of the coercive force.

The curves of magnetization and demagnetization for Fe-28%Cr-7%Co-2%Mo-0.5%Si alloy after all heat treatments are shown in figure 6.

![Figure 6. The curves of magnetization and demagnetization for Fe-28%Cr-7%Co-2%Mo-0.5%Si alloy after (a) – A heat treatment and additional annealing, (b) – B heat treatment and additional annealing.](image3)

The hysteresis loop squareness is about 62 % and was calculated by formula $\eta = \frac{(BH)_{max}}{B_r \times H_c}$. 
The following maximum magnetic properties can be obtained on the alloy: residual induction $B_r$ up to 1.3 T, coercive force $H_c$ up to 40 kAm$^{-1}$ and maximum energy product $(BH)_{max}$ up to 31 kJm$^{-3}$.

4. Conclusion
The powder Fe-28%Cr-7%Co-2%Mo-0.5%Si hard magnetic alloy was investigated. The average coefficient of thermal expansion of the alloy is $13.1\times10^{-6}$ K$^{-1}$ in the temperature range between 20 °C and 900 °C, which is well-matched to that of the commercial Fe-Cr-Co alloy with 15 wt. % of cobalt.

The hardness of the alloy increases from 228 to 250 HV 30 in the temperature range of spinodal decomposition at 600 – 660 °C.

The maximum magnetic properties on the alloy was received after the TMT temperature of 540 °C and was the following: residual induction $B_r$ up to 1.3 T, coercive force $H_c$ up to 40 kAm$^{-1}$ and maximum energy product $(BH)_{max}$ up to 31 kJm$^{-3}$.

It was found that the additional annealing of the alloy till 460 °C can increase the coercive force approximately for 6 kAm$^{-1}$.

Due to the level of the magnetic properties the studied powder magnetic alloy can be used as a material for permanent magnets.

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