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RESEARCH

The Effects of Thermomagnetic Treatment on the Magnetic Properties of Nanocrystalline Fe–O and Fe-Co–O Pressed Compacts

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Abstract
The influence of thermal and thermomagnetic treatment on the magnetic properties of iron—cobalt oxides compacts fabricated by powder metallurgy is studied. The influence of magnetic pulse processing (MPP) on the formation of the phase composition and magnetic properties of nanocrystalline α-Fe (50%) + Fe_2O_3 (50%); α-Fe (50%) + Fe_2O_3 (40%) + Co_3O_4 (10%) and α-Fe (50%) + Fe_2O_3 (30%) + Co_3O_4 (20%) pressed powder compacts during synthesis in a high-energy mill and subsequent annealing have been investigated. According to the X-ray diffraction analysis, annealing α-Fe (50%) + Fe_2O_3 (50%) pressed samples at 250 ℃ in air, promotes the oxidation of α-Fe and FeO to magnetite (Fe_3O_4). Additional annealing of the compact in vacuum at 250 ℃ increases its remnant magnetization and magnetic anisotropy. Whereas, increasing the concentration of Co_3O_4 oxide has no strong effect on the coercivity and residual magnetization of the compacts. Eventually, thermomagnetic treatment of the α-Fe (50%) + Fe_2O_3 (30%) + Co_3O_4 (20%) system does not improve its magnetic properties.

Keywords Powder metallurgy · Fe-Co Powder Compacts · Nanocrystalline powders · Magnetic properties · Magnetic pulse processing · Thermomagnetic processing

1 Introduction

Mechanical milling, one of the first steps during powder metallurgy, has the advantage to obtain a significant amount of ultradispersed materials in one stage [1–8]. Nowadays, ball milling with high energy is used for producing nanocrystalline powders [9–11] by powder metallurgy. Cellular dislocation structures formed inside the powder particles leads to the formation of randomly oriented "blocks", separating each parts by high-angle boundaries, i.e. the nanocrystalline grains [12, 13].

One of the current interests in powder metallurgy method is to obtain new hard magnetic materials with novel properties and at low cost [14, 15]. There is a consensus the most suitable materials for the manufacture of permanent magnets are ferrites [16–18]. However, the price level for barium and strontium ferrite compounds forces manufacturers of magnets to look for cheaper analogues. An alternative could be nanocrystalline magnets based on iron oxides [19] because they are cheap, but their magnetic properties are significantly lower than those for hexaferrite-based ones. Finding ways to increase the magnetic properties of cheap materials based

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on iron oxide is currently an important scientific and challenging problem.

Thermal treatment is one of the most important steps during synthesis for obtaining permanent magnets with a final desired crystalline structure, which in turn, has a significant influence on the magnetic properties [20, 21]. Besides, previous results [22, 23] show that the application of magnetic pulse processing (MPP) has positive effects on the magnetic properties of the resulting permanent magnets. Moreover, the magnetic properties are also influenced by the presence of nanocrystalline phases in the samples [23]. In fact, it has been shown that processing iron ore waste in high-energy mills allows the formation of magnetically hard iron oxide powders with particle size 1 μm and coercivity and remnant magnetization values of 190 Oe and 0.5 kGs, respectively [24].

Cobalt doping of iron oxides, including hematite, can significantly improve their magnetic properties such as coercive force (up to 20–35%) and residual induction (up to 40%) [25, 26].For example, the effect of Co doping on the structural, optical, dielectric and magnetic properties of γ-Fe_2O_3 (maghemite), synthesized by chemical coprecipitation [27], shows that the saturation magnetization (Ms) of maghemite nanoparticles increases by 17% with 5% cobalt doping. This is due to the formation of new magnetic phases with a parallel arrangement of Co and lattice spins in the bulk of maghemite. This positive effect of cobalt on the magnetic properties of iron oxides is considered in the production of permanent magnets which significantly can reduce the production costs.

Previous works using chemical-metallurgical methods showed the possibility of obtaining nanodispersed powders of hematite (α-Fe_2O_3) and maghemite (γ-Fe_2O_3) from rolling mill scales [28, 29]. The main stages of that method consist of hydroxide precipitation with the use of alkali at constant pH, washing, drying, and dehydration. The saturation magnetization of hematite and maghemite at room temperature (64 and 2.2 Am^2/kg, respectively), and the coercive force (232,9 and 474 Oe, respectively) allow them to be used as sorbents of heavy ions in the industry of medicine and agriculture. The purpose of this work is to study the effectiveness of using MPP during heat treatment on the magnetic properties of press compacts based on Fe–O and Fe-Co–O systems.

2 Methodology

Powders of iron oxide α-Fe_2O_3 (hematite) and their mixtures with α-Fe and cobalt oxide Co_3O_4, were used as starting materials: α-Fe (50%) + Fe_2O_3 (50%); α-Fe (50%) + Fe_2O_3 (40%) + Co_3O_4 (10%); α-Fe (50%) + Fe_2O_3 (30%) + Co_3O_4 (20%).

The schematic methodology of sample preparation is presented in Fig. S1 in the supporting material. In brief, the initial powders with appropriate compositions were processed in a high-energy ball mill of planetary type AGO-2U for 1—4 h and then pressed to obtain the compacts. The working bodies of AGO-2U were metal balls with 3—6 mm diameters, made of 100Cr6 steel (ШХ15, in the Russian classification), weighing 200 g each. The ratio of ball mass to powder mass was 10:1. The shaft rotation speed was 960 rpm. Compacting the powders was carried out under pressure with a maximum load of 20 ton using a collapsible titanium mold. The resulting compacts (samples) had the shape of parallelepipeds with dimensions: (10.0 ± 0.2) × (10.0 ± 0.2) × (10.0 ± 0.2) mm^3. A photograph of a typical sample is shown in Fig. 1.

Further, low-temperature annealing (250 ℃) of the obtained compacts was carried out in airless and air

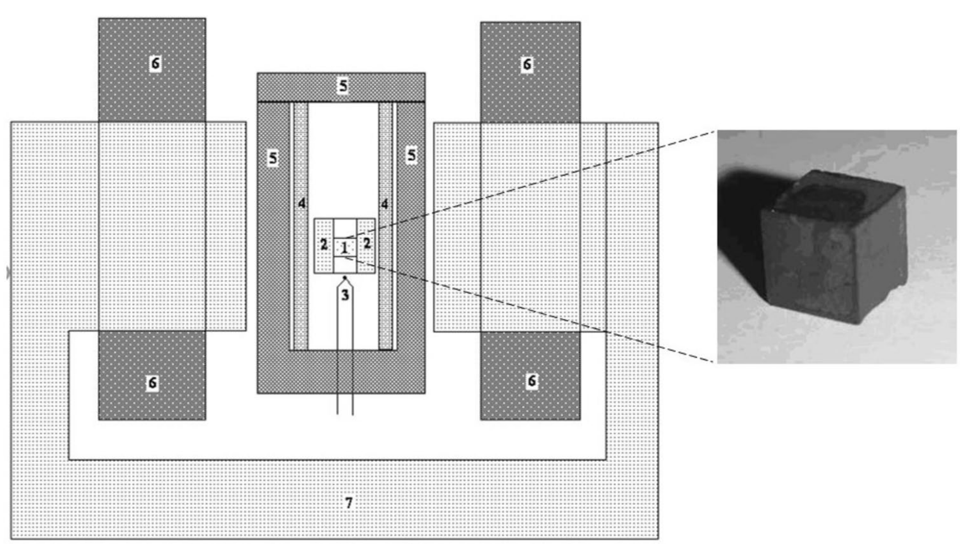

Fig. 1 Schematic representation of the set-up machine used for the thermomagnetic treatment. 1) sample, 2) magnetic flux concentrators, 3) thermocouples, 4) heaters, 5) lining, 6) electromagnet winding, 7) electromagnet yoke. The obtained compacts were parallelepiped in shape with dimensions $(10.0 \pm 0.2)^3$ mm.[3]

atmosphere without and with superimposed magnetic-pulse influence and the magnetic properties were investigated. For the heat and thermomagnetic treatment (TMT) in air, the installation consisted of an electrical resistance furnace and a set-up electromagnet, as shown in Fig. 1. The experiments in vacuum were carried out at a pressure of 10 Pa. The magnetic system consisted of $SmCo_5$ permanent magnets and an arco iron yoke. The magnetic field strength between the concentrators was set to 8,000 kA/m in a 10 mm gap. The temperature was maintained and controlled with an accuracy of ± 2 °C.

X-ray diffraction (XRD) patterns of the samples were obtained on a DRON-4–07 diffractometer. X-ray diffraction analysis was carried out for compacts pressed using a binder (solid oil) and without grease. For this purpose, a special cuvette was used, in which the powder was compacted with the addition of alcohol. X-ray diffraction analysis of powders without a binder made it possible to avoid the appearance of extra lines (from the binder) and reduce the background level, which increased the sensitivity and accuracy of the analysis. The measurement conditions were as follow: Co-Kα radiation (radiation wavelength $\lambda_{K\alpha av} = 0.179$ nm (1.79 Å)); tube operating mode $U = 40$ kV, $I = 30$ mA. The slots were: 8 mm (from the tube), 1 mm (reception on the circumference of the goniometer), 1 mm (in front of the counter); monochromator C (graphite). The range of diffraction angles 2θ varied from 20° to 120°; the shooting step was 0.1°; exposure per shooting point was 3 s. Quantitative phase analysis was carried out using the X-ray program PHAN%. This method of quantitative phase analysis is a modification of the Rietveld method, based on minimizing the difference between the experimental and model (calculated) diffractogram taken point by point [30].

3 Results and Discussion

Figure 2 shows XRD results of the samples. Figure 2(a) shows the patterns for the mixtures α-Fe (50%) + Fe_2O_3 (50%), α-Fe (50%) + Fe_2O_3 (40%) + Co_3O_4 (10%), and α-Fe (50%) + Fe_2O_3 (30%) + Co_3O_4 (20%) after 4 h mechanoactivation (grinding) in a high-energy ball mill of planetary type AGO-2U. The phase composition and the calculated average sizes of nanocrystallites of the resulting mixtures are given in Table 1. The table shows that increasing the cobalt content in the mixture leads to an increase in the amorphous phase (APh), i.e., the most amorphous phase is formed in the initial sample containing Co_3O_4 (20%). It is also evident that in all samples containing α-Fe (50%) in the initial state, the ratio of the amount of α-Fe to FeO is almost unchanged.

During heating the compacts, different mechanism and kinetics transformations are expected to occur. This is reflected in the resulted phase composition of the compacts after heating in air and in vacuum (10 Pa) at 250 °C. Heat treatment in air was carried out on the press-compacts with initial composition α-Fe (50%) + Fe_2O_3 (50%). The X-ray diffraction analysis of the sample in the initial state containing α-Fe (50%) + Fe_2O_3 (50%) was carried out after 1, 2 and 5 h of annealing the compact in air and they are shown in Fig. 2(b). After 1 h annealing, Fe_3O_4 oxide lines appear on the X-ray diffraction pattern. After 2 h annealing, the FeO phase is not more detected in the samples. With further annealing (5 h), the diffraction pattern does not undergo significant changes. Figure 1(c) shows the dependence of changes in the phase composition of the sample α-Fe (50%) + Fe_2O_3 (50%) with annealing time. It can be seen that the amount of FeO for the first 2 h sharply drops to zero, and the amount of α-Fe decreases significantly from 42 to 30%. At the same time, the Fe_3O_4 phase appears with 70% amount after 2 h annealing. During the next 3 h annealing, the amount of α-Fe and Fe_3O_4 remained practically unchanged. As a result of annealing, the structure of the sample changed and sintering of the powders occurred. It is obvious that in the first 2 h annealing, α-Fe and FeO are oxidized by oxygen in the air to form Fe_3O_4. After 2 h annealing, the sample is completely sintered and oxidation processes are practically absent due to the difficulty of oxygen diffusion into the sample.

The XRD analysis of powder mixtures with compositions α-Fe (50%) + Fe_2O_3 (50%), α-Fe (50%) + Fe_2O_3 (40%) + Co_3O_4 (10%) and α-Fe (50%) + Fe_2O_3 (30%) + Co_3O_4 (20%), following high-energy heat treatment for 4 h reveals the formation of α-Fe, FeO and amorphous phase. It can be assumed that the amorphous phase is formed by the reaction α-Fe + FeO → APh. On the other hand, cobalt oxide possibly acts as a catalyst for solid-phase reactions occurring in this system, with other effects such as, accelerating the diffusion of components. This is evidenced from Table 1 that an increase in the concentration of cobalt in the mixture (0–10%–20%) leads to a corresponding increase in the proportion of amorphous phase (22%—28%—35%).

It can be seen that with increasing annealing time, the XRD peaks in Fig. 2(b) become sharp, which means that the crystallization of the samples improves. The increase in crystallinity in the powder compacts can be likely explained by the fact that during annealing the areas of coherent scattering increase, as well as by reducing the level of microdistortion. Note that the FeO and the amorphous phase are weakly magnetic and are thermodynamically unstable at room temperature. Therefore, milling the α-Fe (50%) + Fe_2O_3 (50%) composition in a high-energy mill (grinding for 4 h) and subsequent low-temperature annealing (250 ℃) promotes the metastable phases to decompose into stable ferromagnetic α-Fe and Fe_3O_4 during the first 2 h of annealing, which is also confirmed by the data presented in [11, 31].

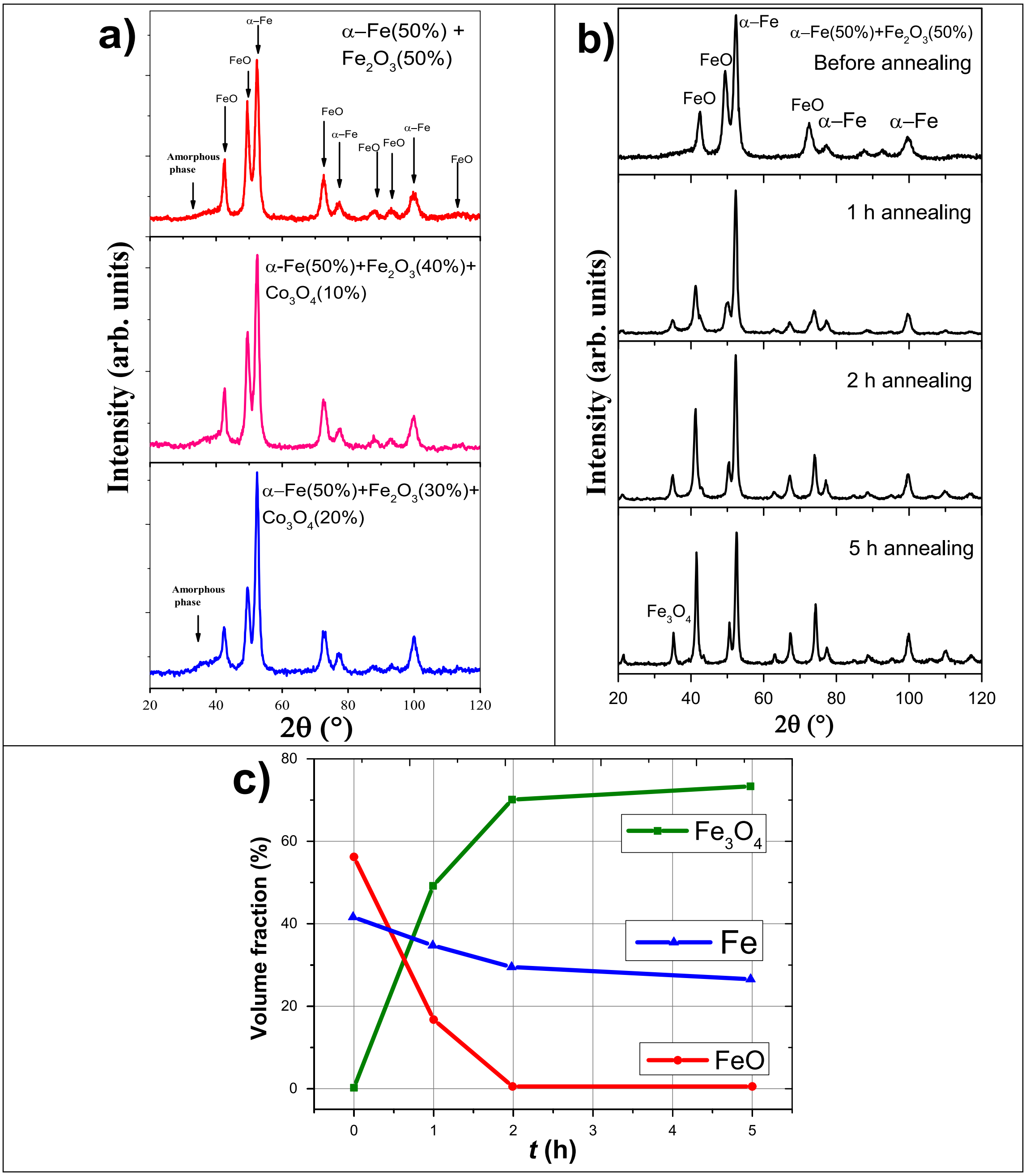

Fig. 2 **a** XRD patterns of powder mixtures α-Fe (50%) + Fe_2O_3 (50%), α-Fe (50%) + Fe_2O_3 (40%) + Co_3O_4 (10%) and α-Fe (50%) + Fe_2O_3 (30%) + Co_3O_4 (20%) subjected to high-energy treatment (grinding) for 4 h. **b** XRD diffraction patterns of a sample with an initial composition of α-Fe (50%) + Fe_2O_3 (50%) before and after its annealing at 250 °C. **c** Change in the phase composition of a sample with the original composition α-Fe (50%) + Fe_2O_3 (50%) after annealing at 250 °C in air

Experiments were also conducted to study the influence of external field and annealing conditions, in vacuum and air at 250 ℃, on the magnetic properties of the compacts with initial composition α-Fe (50%) + Fe_2O_3 (50%), which has been mechanically activated for 4 h. At the same time, the external magnetic field was applied in two mutually perpendicular directions relative to the compact. Figure 3 shows the dependences of the magnetization on the magnetic field strength for the α-Fe (50%) + Fe_2O_3 (50%) sample. Note that annealing in vacuum for 5 h increases the residual magnetization of the compact more than 1.5 times. One of the reasons for this improvement in the magnetic properties of the sample is that during vacuum annealing, iron-containing powders

Table 1 Phase composition of powder and sizes of nanocrystalline blocks after 4 h grinding

Original composition	Phase composition	Volume fraction (%)	<*D*> (nm)
α-Fe (50%) + Fe_2O_3 (50%)	α-Fe	36	9
	FeO	42	14
	APh	22	-
α-Fe (50%) + Fe_2O_3 (40%) + Co_3O_4 (10%)	α-Fe	33	10
	FeO	39	14
	APh	28	-
α-Fe (50%) + Fe_2O_3 (30%) + Co_3O_4 (20%)	α-Fe	32	10
	FeO	33	10
	APh	35	-

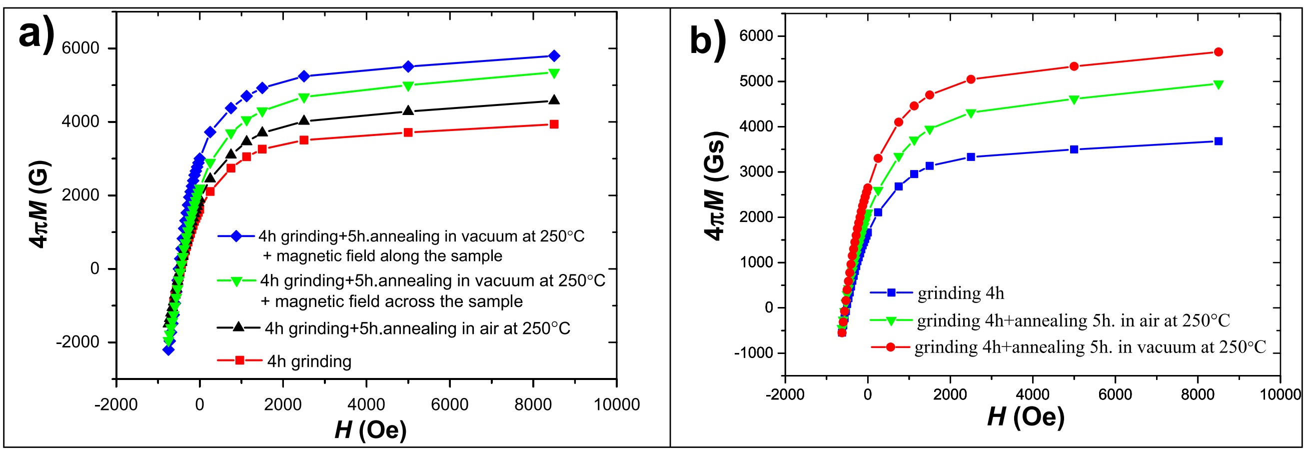

Fig. 3 **a** Magnetization of the α-Fe (50%) + Fe_2O_3 (50%) press compact on the magnetic field strength. **b** Dependence of magnetization on the external field for the sample α-Fe (50%) + Fe_2O_3 (40%) + Co_3O_4 (10%)

are cleaned of some dissolved impurities, such as carbon, oxygen, silicon atoms, etc.

Comparing the obtained data with previous works [11, 31–33] indicates that phase transformations in bulk during annealing are not very different from those occurring in powders. The main distinguishing feature is the larger amount of residual iron in the pressed compacts due to the difficulty of oxygen diffusion deep into the sample.

Figures 4(a) and (b) shows the dependence of the magnetic properties on the time of annealing in vacuum of pressed powder compacts with initial composition α-Fe (50%) + Fe_2O_3 (50%). The graphs show that saturation magnetization and residual magnetization reach saturation with increasing annealing duration, and the coercivity and maximum energy pass through the maximum (6 h). It suggests that for alloys with this composition, the optimum heat treatment is in a vacuum furnace at 250 °C for 6 h.

Table 2 lists the values of saturation magnetization, remanent magnetization, coercive force and magnetic field energy after annealing in air and in vacuum at a temperature of 250 ℃ for the sample α-Fe (50%) + Fe_2O_3 (50%). Comparing the magnetic properties of the α-Fe (50%) + Fe_2O_3 (50%) powder compact subjected and not subjected to magnetic-impulse treatment shows that TMT allows to raise the value of residual magnetization by 24%, and at the same time, it reduces the coercivity by 6%. Note from the values in the table that annealing in vacuum is not effective for increasing the magnetic properties of the alloys in the compacts. Therefore, in order to investigate the effect of TMT on the magnetic properties of the other samples, the process was carried out in air. The change of magnetic properties passes through the maximum, which is observed at 6 h of treatment at 250 °C, regardless of the presence or absence of vacuum or magnetic-pulse influence.

Figures 4(c) and (d) show the dependence of the coercivity and residual magnetization on the annealing time and TMT in air for the sample with initial composition α-Fe (50%) + Fe_2O_3 (30%) + Co_3O_4 (20%). It follows from the graph that it is not reasonable to carry out TMT on the compacts with Fe-Co–O alloys to increase its magnetic properties. It is recommended to use TMT for the treatment of powder systems, where in most cases a positive effect is

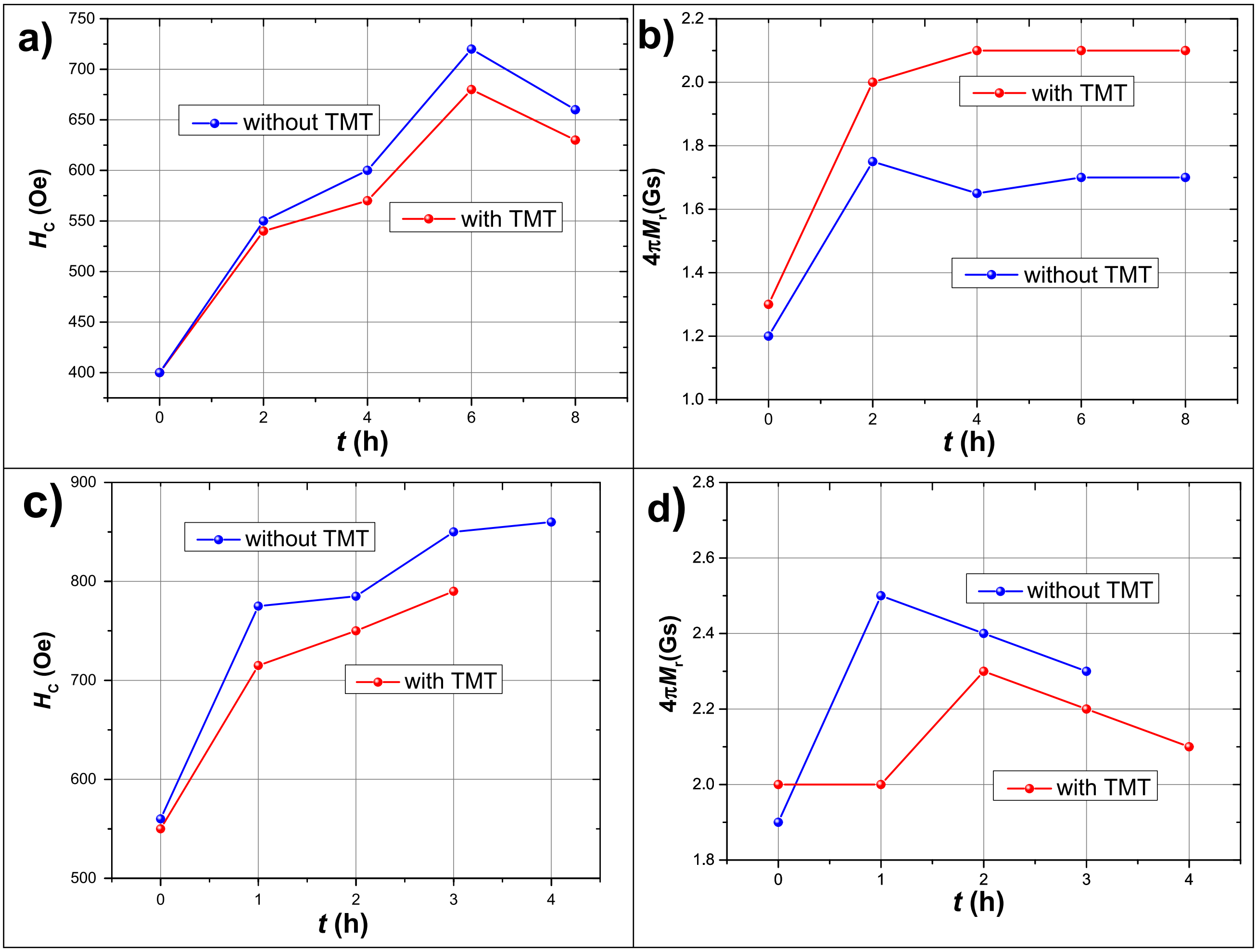

Fig. 4 Dependences of coercivity (H_c) and residual magnetization ($4\pi M_r$) on annealing time and TMT: **a** and **b**—for α-Fe (50%) + Fe_2O_3 (50%) samples in vacuum; **d** and **c**—for α-Fe (50%) + Fe_2O_3 (30%) + Co_3O_4 (20%) samples in air

Table 2 Magnetic properties of a compact sample made by powder metallurgy of composition 1, (α-Fe (50%) + Fe_2O_3 (50%)), after annealing in air and in vacuum at 250 °C

In air				In vacuum			
Without TMT		With TMT		Without TMT		With TMT	
Hc (Oe)	4π*Mr* (кG)	H_c (Oe)	$4\pi M_r$ (кG)	H_c (Oe)	4π*Mr* (kG)	*Hc* (Oe)	4π*Mr* (kG)
400	1,2	400	1,3	400	1,2	400	1,3
550	1,75	540	2	568	1,75	537,5	2,05
600	1,65	570	2,1	600	1,65	568	2,1
720	1,7	680	2,1	712,5	1,7	681,25	2,075
660	1,7	630	2,1	662,5	1,7	625	2,1

observed. Thus, it should be stated that the application of TMT does not allow increasing the magnetic properties of Fe–O-based compacts, but there is a visible tendency to decrease the deviations of values, which are observed during treatment in air and vacuum. This allows us to conclude that TMT is preferred if the task is to obtain alloys with controlled magnetic properties.

The next stage of the work was to study the influence of Co_3O_4 additives in the initial matrix, before high-energy treatment, on the magnetic properties of the obtained compacts. Cobalt oxide does not possess significant magnetic properties, and its addition was conditioned pursuing the formation of magnetic ferrite, $Fe_{3-x}Co_xO_4$. It has been discussed above that probably all cobalt passes are in the amorphous

Table 3 Effect of Co_3O_4 additives on magnetic properties of pressed powder compacts

Samples	α-Fe (50%) + Fe_2O_3 (50%)		α-Fe (50%) + Fe_2O_3 (40%) + Co_3O_4 (10%)		α-Fe (50%) + Fe_2O_3 (30%) + Co_3O_4 (20%)	
	grinded	post-annealed	grinded	post-annealed	grinded	post-annealed
H_C (kOe)	0,57	0,68	0,56	0,78	0,57	0,76
4π*Mr* (kG)	3,6	4,8	4	5,49	4,3	5,6
4π*Ms,* (kG)	6,99	9,52	7,66	9,91	8,14	9,94

phase, since the diffractograms (Fig. 2(a)) lack of peaks corresponding to the Co_3O_4 and $Fe_{3-x}Co_xO_4$ phases. The heat treatment time was 6 h.

Figure 4(d) shows the magnetization dependence of the compacts composed of α-Fe (50%) + Fe_2O_3 (40%) + Co_3O_4 (10%) with annealing time. Table 3 lists the values of the main magnetic terms of the studied compacts depending on the cobalt concentration and processing conditions. The figure reveals that additional annealing of the samples after high-energy grinding improves their magnetic properties. In particular, the saturation magnetization and remnant magnetization increase, while the values of coercivity remain unchanged. Whereas, annealing in vacuum is preferable than annealing in air. On the other hand, experiments have shown that for the system α-Fe (50%) + Fe_2O_3 (30%) + Co_3O_4 (20%) this dependence is broken. The increase of cobalt concentration leads to the fact that the efficiency of vacuum annealing becomes lower than annealing in air.

From the results listed in Table 3, it is evident that Co_3O_4 additions contribute to the increase of the magnetic properties, but not as strong as expected. For example, in reference [34], powders with an initial composition of Fe (35%) + Co (30%) + Fe_2O_3 (35%) and Fe (43%) + Co (22%) + Fe_2O_3 (35%) show that cobalt additives do not affect the phase composition of powders after grinding; however, the transformations slow down and the processing time doubles (up to 6 h). After grinding and at low-temperature vacuum annealing, the mixture containing 30% Co compared to the original alloy has a 15% greater residual magnetization and a 30% greater magnetic energy, respectively, ($4\pi M_r = 6{,}0$ kG, $(BH)_{max} = 6{,}08$ kJ/m^3), while maintaining a high level of coercive force ($H_c = 282.74$ Oe) [34].

4 Conclusions

Mixtures of Fe—O and Fe—Co—O powders were consolidated into compacts by powder metallurgy and their crystalline and magnetic properties were studied. In high-energy mill conditions, an amorphous phase is formed, which increases with increasing grinding time and amount of cobalt. The XRD of the treated samples do not show peaks of cobalt- phases, indicating the transition of all or most of the cobalt to the amorphous phase. Co_3O_4 additives do not have a strong effect on the values of coercivity but increase the values of residual magnetization. The use of TMT to increase the magnetic properties of press compacts based on Fe—O and Fe—Co—O alloys is not advisable. The use of TMT, in most cases, makes it possible to level out the difference in the values of the magnetic properties of press compacts annealed in air and vacuum. This effect can be used to create magnetic materials with specified properties.

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Authors Contribution Data acquisition, methodology, data curation and formal analysis (A.S. Lileev, J. Kargin, Y.V. Konyukhov, DG Zhukov and H. Sanchez Cornejo), conceptualization, interpretation, validation, data curation and visualization (Ji Won Seo, S.N. Holmes, J. Albino Aguiar, C.H.W. Barnes and L. De Los Santos Valladares) and all authors have contributed to write and correct the manuscript.

Data Availability No datasets were generated or analysed during the current study.

Declarations

Competing Interests The authors declare no competing interests.

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