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STUDY OF THE EFFECT OF HAFNIUM ON THE MAGNETIC PROPERTIES OF PERMANENT MAGNETS WITH SINGLE-CRYSTAL STRUCTURE FROM ALLOY YuNDKT5AA

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The causes of the action of hafnium additives on the magnetic properties of single-crystal permanent magnets from alloy YuNDKT5AA are studied. The temperature regimes of the isothermal magnetic treatment of single-crystal permanent magnets from alloy YuNDKT5AA with a hafnium additive is corrected. A new mode of multistage tempering is suggested and an operation of thermal stabilization of permanent magnets is introduced.

Key words: alloying, hafnium, single-crystal magnets, isothermal magnetic treatment, tempering, magnetic properties, thermal stabilization, temperature coefficient of induction.

INTRODUCTION

Single-crystal permanent magnets produced from alloy YuNDKT5AA possess the highest level of magnetic properties and the lowest value of the temperature coefficient of induction (TCI) of all the other magnets produced from alloys of type YuNDK and YuNDKT. For this reason single crystals magnets from alloy YuNDKT5AA are used to make high-duty electrical machines, devices and control systems.

The chemical composition of alloy YuNDKT5AA and the magnetic properties of single-crystal permanent magnets fabricated from the former are specified by the GOST 17809–72 Standard and are presented in Table 1.

We have shown in our previous works that further improvement of the magnetic and operating properties of single-crystal permanent magnets with respect to the recent level can be provided by introducing up to 1% metallic hafnium into the composition. Hafnium raises the susceptibility of alloy YuNDKT5AA to formation of single-crystal structure upon growth, which may increase the efficiency of the process of growth of single crystals of this alloy by more than a factor of 3 (Fig. 1). The level of magnetic properties of permanent magnets obtained from these single crystals does not decrease substantially and still matches the range standardized by GOST 17809–72 [2 – 4].

In the present work we made an attempt to determine the causes of the growth of the magnetic properties of single-crystal permanent magnets from alloy YuNDKT5AA upon the addition of hafnium. The final aim of the work was to raise the magnetic and operating properties of single-crystal permanent magnets from alloy YuNDKT5AA above the recent level.

METHODS OF STUDY

We melted two compositions based on the hard magnetic alloy YuNDKT5AA. The first chemical composition matched fully the prescription of GOST 17809–72. The second alloy contained an additive of 1 wt.% hafnium. Hafnium was introduced into the metal at a corresponding decrease in the content of iron.

The blend material was composed of pure metals of the following grades: cobalt K-0 (GOST 123–98), nickel N-0 (GOST 849–97), copper M-0 (GOST 859–2001), aluminum A99 (GOST 11069–2001), titanium iodide (TU 48-4-282–73), refined carbonyl iron (ChMTU-1-8884–70), sulfur (GOST 127.1–93), and hafnium iodide (GOST 22517–77).

The alloys were melted in an ISV-0.016 vacuum induction furnace with a lining from alumina. The process was conducted as follows: cobalt, iron and nickel were put into the crucible and copper, titanium, sulfur (in the form of iron

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TABLE 1. Chemical Composition and Magnetic Properties of Single-Crystal Permanent Magnets from the Base Hard Magnetic Alloy YuNDKT5AA

Alloy	Content of elements, wt.%							Magnetic properties		
	Co	Ni	Al	Ti	Cu	S	Fe	B_r, T	H_c, kA/m	$(BH)_{max}$, kJ/m^3
YuNDKT5AA (GOST 17809–72 [1])	34.5 – 35.5	14.0 – 14.5	7.0 – 7.5	5.0 – 5.5	2.5 – 4.6	0.1 – 0.2	Rem.	1.05 – 1.1	115 – 120	80.0 – 88.0

sulfide), aluminum and hafnium were put into the batcher. The blend was degassed in vacuum (1.33×10^{-2} Pa), the chamber was filled with high-purity argon (99.99%), and then the melting was performed. The temperature of heating of the melt before casting was 1650°C [5, 6]. The ready melt was cast into baked ceramic molds fabricated by the method of investment patterns. The cast preforms had a coarse-grain polycrystal structure.

The content of the main and alloying elements was controlled with the help of x-ray and wave dispersion spectrometers of types SRM-25M (Russia) and ARL ADVANT’X (Switzerland), respectively. The content of sulfur and carbon was determined using an AS-7932, AN-7529 (Russia) and ELTRA CS-800 (Germany) rapid analyzers.

Single-crystal preforms were fabricated by recrystallization of the polycrystalline preforms by the method of Bridgman controlled unidirectional hardening in “Kristallizator-203” devices with induction heating on a seed in refractory containers from pure alumina [4, 5]. The rate of growth of the single crystals (the speed of displacement of the heater) was 0.4 mm/min in all the cases.

The thermomagnetic treatment of the grown preforms after a preliminary mechanical treatment was performed with the help of PVK-1.4-17 and PKL-1.2-36 resistance furnaces, an isothermal bath, and a special electromagnet in the following way: at first, the preform was heated (in the PVK-1.4-17 furnace) to 800°C at a rate of 100 K/h, held at this temperature for 60 min, placed into the PVK-1.4-17 furnace heated preliminarily to 1250 ± 2°C, and held at this temperature for 15 min. Then the preform was cooled for 75 sec in a magnetic field with intensity no lower than 250 kA/m in air, transferred into the isothermal bath heated to 805 ± 1°C, held in a magnetic field with intensity no lower than 250 kA/m, and then transferred into the PKL-1.2-36 furnace heated to 500°C. After the thermomagnetic treatment of the whole of the batch of magnets, the PKL-1.2-36 furnace was switched to the mode of tempering.

The tempering was performed by two variants. The first variant matched fully the GOST 17809–72 Standard for alloy YuNDKT5AA, i.e., consisted of two stages and lasted for 25 h in total. The second variant was developed specially for our study and consisted of four stages with total duration of 10.5 h. The variants and the stages of the tempering are presented in Table 2.

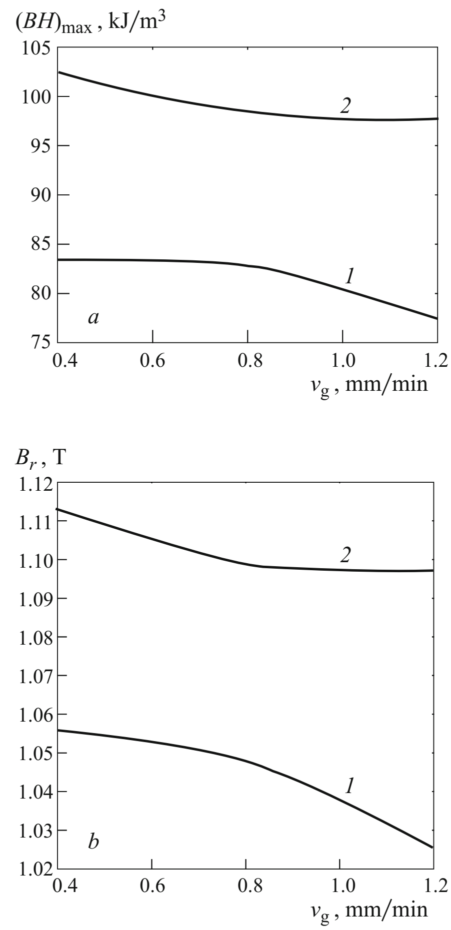

Fig. 1. Dependence of the maximum energy product $(BH)_{max}$ (*a*) and residual magnetic induction B_r (*b*) of permanent magnets on the rate of growth of the single crystals v_g [4]: *1*) alloy YuNDKT5AA; *2*) alloy YuNDKT5AA + 1% hafnium.

TABLE 2. Variants and Regimes (Stages) of Tempering of Single-Crystal Preforms

Variants of tempering	Regimes (stages) of tempering	Temperature, °C	Time, h
First (two-stage tempering)	1st stage	650	5.0
	2nd stage	560	20.0
Second (four-stage tempering)	1st stage	680	0.5
	2nd stage	650	2.0
	3rd stage	580	3.0
	4th stage	560	5.0

TABLE 3. Results of Chemical Analysis of Alloys YuNDKT5AA

Alloy	Content of elements, wt.%								
	Co	Ni	Al	Ti	Cu	Hf	S	C	Fe
YuNDKT5AA without hafnium	35.39	14.26	7.12	5.36	3.97	–	0.14	0.027	Rem.
YuNDKT5AA with 1% hafnium	35.27	14.38	7.18	5.24	4.01	0.98	0.12	0.013	Rem.

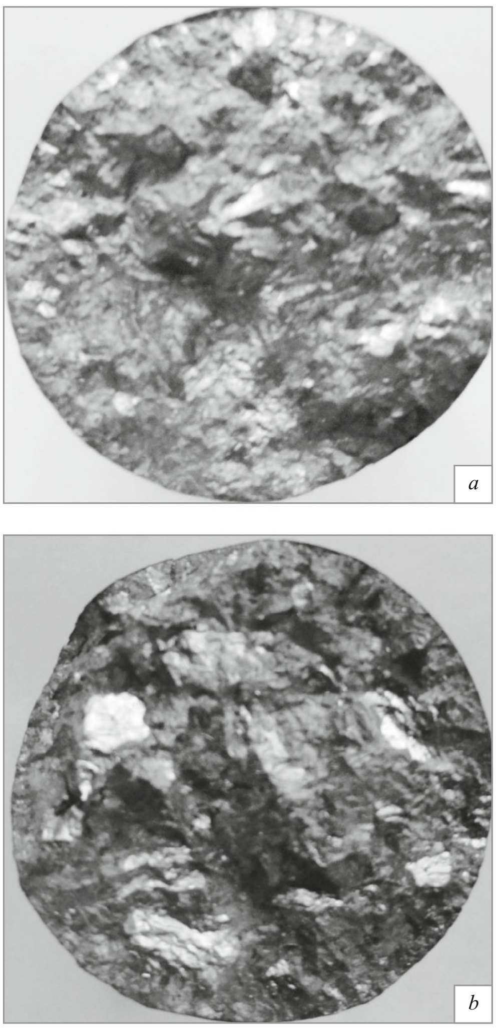

Fig. 2. Microstructure of polycrystalline preforms 19 mm in diameter from alloys YuNDKT5AA without hafnium (*a*) and with an additive of 1% hafnium (*b*), × 1.5.

TABLE 4. Magnetic Properties of Single-Crystal Permanent Magnets

Alloy	Tempering regime	B_r, T	H_{cb}, kA/m	$(BH)_{max}$, kJ/m^3
YuNDKT5AA	Two-stage	1.048	120.08	84.57
	Four-stage	1.057	118.93	86.16
YuNDKT5AA with 1% hafnium	Two-stage	1.088	120.07	88.90
	Four-stage	1.113	124.47	102.46

When the tempering was finished, the preforms were cooled to room temperature together with the furnace.

The quantitative phase analysis was performed with the help of a D8 ADVANCE x-ray diffractometer (Germany).

Thermal stabilization of the magnets was performed in a resistance furnace at 100°C for 10 h in air.

The magnetic properties of the ready magnets were controlled using a PERMAGRAPH C-300 device (Germany) by plotting the demagnetization curves.

RESULTS AND DISCUSSION

The chemical composition of alloys YuNDKT5AA without hafnium and with 1.0% hafnium is presented in Table 3.

The conditions of fabrication of polycrystalline preforms from the studied alloys were identical. Analysis of the microstructure has shown that the introduction of hafnium into the composition increases the grain size in the polycrystalline preforms (Fig. 2). This means that the susceptibility of these preforms to formation of a single-crystal structure under unidirectional hardening has increased [2, 3, 5].

The polycrystalline preforms were used to produce single-crystal preforms by the known method. The perfection of the single-crystal structure of the grown preforms was estimated by the metallographic method. It was shown that the single-crystal preforms fabricated from the alloy with hafnium had a more perfect structure than the preforms without hafnium.

The single-crystal preforms were subjected to isothermal treatment and tempering by the first and second variants (Table 3) and then used to make permanent magnets. The magnetic properties of the magnets are presented in Table 4.

It can be seen from Table 4 that the introduction up to 1% hafnium into alloy YuNDKT5AA causes growth of all the magnetic properties of the single-crystal permanent magnets. The four-stage tempering regime is preferable, because it provides higher magnetic properties, especially in the magnets with hafnium.

To determine the causes of such effect of hafnium on the magnetic properties we performed a quantitative phase analysis of the alloys after an isothermal treatment followed by four-stage tempering. The results of the analysis in the form of deciphered diffractograms are given in Fig. 3. The alloys contain α- and α′-phases. Phase α has a bcc lattice, its structure is similar to AlNi, and the lattice parameter is 0.2885 nm.

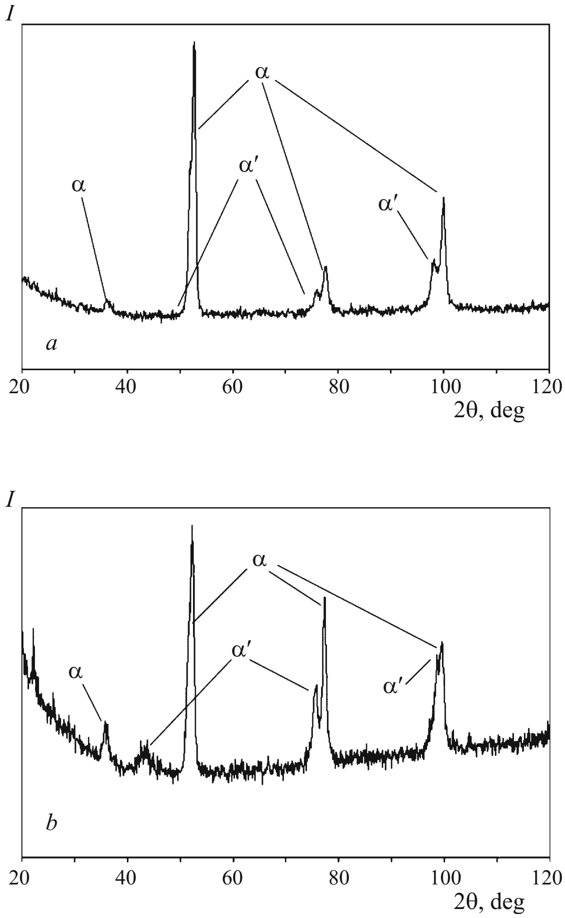

Fig. 3. Diffractograms of single-crystal permanent magnets from alloys YuNDKT5AA without hafnium (*a*) and with an additive of 1% hafnium (*b*) after an isothermal treatment followed by four-stage tempering: *a*) α = 60%, α′ = 40%; *b*) α = 70%, α′ = 30%.

Phase α′ also has a bcc lattice, its structure is similar to AlFe, and the lattice parameter is equal to 0.2909 nm.

It can be seen from Fig. 3 that the introduction of up to 1% hafnium into alloy YuNDKT5AA increases the content of the strongly magnetic α′-phase in the structure by almost 10%. This seems to be the main reason behind the growth in the magnetic properties of the single-crystal permanent magnets.

Another possible cause of elevation of the magnetic properties is the higher perfection of the single-crystal structure of the grown preforms due to the enhancement of the susceptibility of alloy YuNDKT5AA to single crystal growth upon unidirectional hardening as a result of the introduction of hafnium. The positive influence of hafnium on the susceptibility of alloys of type YuNDKT to single crystal growth is shown in [2, 3, 5]. An indirect evidence of elevation of the susceptibility of alloy YuNDKT5AA to single crystal growth is the growth of the grain size in the polycrystalline preforms after the introduction of hafnium (Fig. 2).

A very important property of single-crystal permanent magnets is their high temperature stability. On the one hand, it is a result of the absence of grain boundaries in single-crystal magnets, and, on the other hand, it is explainable by the morphological stability of the phases. We judge on the temperature stability of permanent magnets by the value of the TCI. The higher the absolute value of the TCI the higher the temperature stability of the permanent magnet. We have established that the single-crystal permanent magnets fabricated from alloy YuNDKT5AA with 1% hafnium have TCI 30 – 50% lower than those from the alloy without hafnium after the same treatment involving an isothermomagnetic treatment, a four-stage tempering, and a thermostabilization treatment at 100°C for 10 h.

CONCLUSIONS

The main cause of elevation of the magnetic properties of single-crystal permanent magnets from alloy YuNDKT5AA with an addition of up to 1% hafnium is growth of the content of the strongly magnetic α′-phase. Another cause is improvement of the perfection of the single-crystal structure of the grown preforms due to the increased susceptibility of the alloy with hafnium to formation of a single-crystal structure in castings that undergo unidirectional hardening. In addition to raising the principal magnetic characteristics the addition of hafnium into alloy YuNDKT5AA lowers the value of the TCI in single-crystal permanent magnets by 30 – 50%.

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