



EFFECT OF NIOBIUM, TITANIUM AND MOLYBDENUM ADDITIONS TO $\text{Sm}_2\text{Fe}_{17}$ OBTAINED BY MECHANICAL ALLOYING

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ABSTRACT

Results of experimental studies influence of the complex alloying with titanium, molybdenum and niobium on the Curie Temperature (T_C), and magnetism of $\text{Sm}_2\text{Fe}_{17}$ alloy are presented. It is shown, that alloying of $\text{Sm}_2\text{Fe}_{17}$ with Ti, Mo and Nb leads to increase volume and lattice parameters without modification of the symmetry lattice. It was found that complex alloying increases the Curie temperature from 139 °C (initial alloy) to 206 °C. During measurement of the hysteresis properties, it was revealed that alloying with titanium, molybdenum and niobium leads to the widening of the hysteresis loop.

Keywords: mechanical alloying, magnetic material, magnetism, Sm-Fe system, curie temperature.

1. INTRODUCTION

During investigation and development of hard-magnetic materials the main attention of researchers over the last years on the system $\text{Nd}_2\text{Fe}_{14}\text{B}/\alpha\text{-Fe}$ and its additional alloying [1-9] was directed. However, as it's known, magnets based on Nd-Fe-B systems can be used only in the limited temperature range: 130 °C to +200 °C. The ceiling limit is due to with the relatively low Curie temperature (312 °C) of the intermetallic compound: $\text{Nd}_2\text{Fe}_{14}\text{B}$ and the bottom limit - with spin-orientation phase transition $\text{Nd}_2\text{Fe}_{14}\text{B}$, which is observed at temperatures below - 131 °C [10].

In this regard, intermetallic compounds based on rare earth element were investigated. Among iron alloys with rare earth elements R_2Fe_{17} -compounds (R – rare earth elements) have the highest iron content and, therefore, the highest saturation intensity. Nevertheless, the R_2Fe_{17} compounds cannot be directly used for manufacturing permanent magnets by reason of the low Curie temperature and plane magnetic crystal anisotropy. Coercive forces of magnetization and Curie temperature (T_C) of R_2Fe_{17} -compounds are usually increase by alloying: Al, Cu, Ga, Ti, Nb, Mo, V, Re, W, Be, etc.

In ones of the first works for the alloying of $\text{Sm}_2\text{Fe}_{17}$ alloy with niobium Sinan *et al.* [11, 12] have shown influence of Nb content on the phase composition, lattice parameter and Curie temperature. In the work [13] the alloy of Sm-Fe-Nb system with a Curie temperature of 230 °C was received, the increase in magnetic characteristics also was noted. It is shown, that alloy $\text{Sm}_{10.2}\text{Fe}_{85.8}\text{Nb}_4$ has the maximum Curie temperature. In the work [14] shown, that in $\text{Sm}_2\text{Fe}_{17-x}\text{Nb}_x$ compound at $x = 0,5 - 1$ the coercive force and remaining magnetization is increase, but when 'x' exceeds 2 the magnetic characteristics worsen. Saito *et al.* investigated the Sm-Fe-Ti system in his works [15, 16]. He showed, that it is possible to increase the Curie temperature of $\text{Sm}_2\text{Fe}_{17}$ alloy to 240 °C by alloying with titanium. At that point, the coercive force increases to 30 kA/m and due to additional heat treatment to 240 kA/m.

In spite of the huge number of works, which are devoted to investigation of the influence of alloying on the magnetic properties of the $\text{Sm}_2\text{Fe}_{17}$ alloy, the compounds still remain insufficiently investigated, although they are very interesting objects for the physics of magnetic phenomena and technology. In this regard, the investigation of the influence complex alloying of the $\text{Sm}_2\text{Fe}_{17}$ with titanium, molybdenum and niobium on the Curie temperature T_C and magnetic properties is of high interest.

2. MATERIALS AND METHODS

Sm-Fe-Nb-Ti and Sm-Fe-Nb-Mo-based alloys of various compositions ($\text{Sm}_2\text{Fe}_{16}\text{Nb}_{0.5}\text{Ti}_{0.5}$, $\text{Sm}_2\text{Fe}_{15.7}\text{Nb}_{0.8}\text{Ti}_{0.5}$, $\text{Sm}_2\text{Fe}_{15.5}\text{NbTi}_{0.5}$, $\text{Sm}_2\text{Fe}_{16}\text{Nb}_{0.5}\text{Mo}_{0.5}$, $\text{Sm}_2\text{Fe}_{15.7}\text{Nb}_{0.8}\text{Mo}_{0.5}$, $\text{Sm}_2\text{Fe}_{15.5}\text{NbMo}_{0.5}$) were prepared by mechanical alloying of initial powders components using Pulverisette-4 planetary mill according to the technique described in [17]. Powders of iron ($d_{90} < 300 \mu\text{m}$), samarium ($d_{90} < 700 \mu\text{m}$), niobium, molybdenum and titanium with a purity of 99,9% were used as the initial components. The duration of the mechanical alloying process was 40 hours, rotation velocity - 400 rpm. To prevent oxidation of the initial powder materials, preparation of powders for mechanical alloying and loading into a mill chamber was carried out in glove box in a nitrogen atmosphere. The obtained powders were annealed in vacuum at a temperature of 660 °C for 30 minutes.

Phase composition was studied by X-ray diffraction on Bruker D8 Advance in $\text{CuK}\alpha$ -radiation ($\lambda = 1,5418\text{\AA}$). Research of the structure and distribution of elements in the volume of powder particle was carried out using scanning electronic microscope Mira 3 Tescan with the Oxford INCA Wave 500 add-on device. Curie temperature (T_C) was determined by the method described in work [21]. Measurements of magnetic properties were performed on a vibration magnetometer VSM 7410 Lake Shore in the range of field induction from 22000 to -22000 Oe (2.2;-2.2 T) in the sensitivity range of ($\pm 4 \text{ emu}$).



3. RESULTS AND DISCUSSIONS

Sm-Fe-Nb-Ti and Sm-Fe-Nb-Mo-based powder alloys obtained by mechanical alloying have fragmented form with particle size $d_{50} = 10 \mu\text{m}$ (Figure-1). SEM

researches have shown that alloying elements are uniformly distributed in the volume of powder and match the chemical makeup of the initial composition (Figure-2).

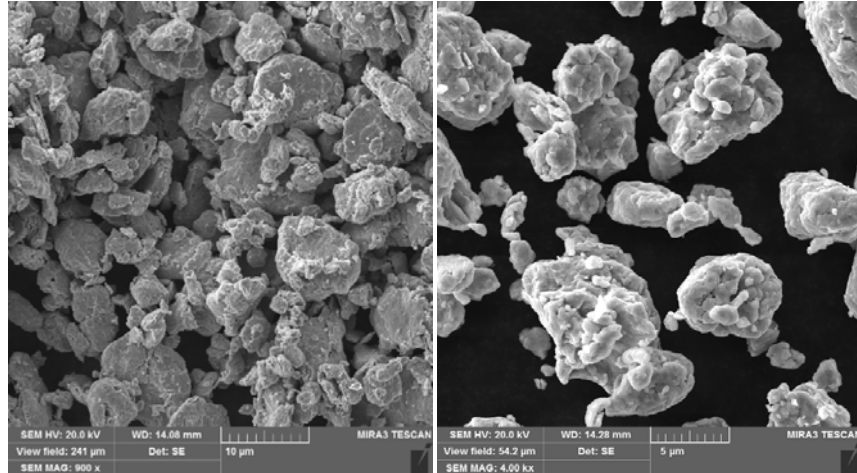


Figure-1. Morphology of powder obtained by mechanical alloying.

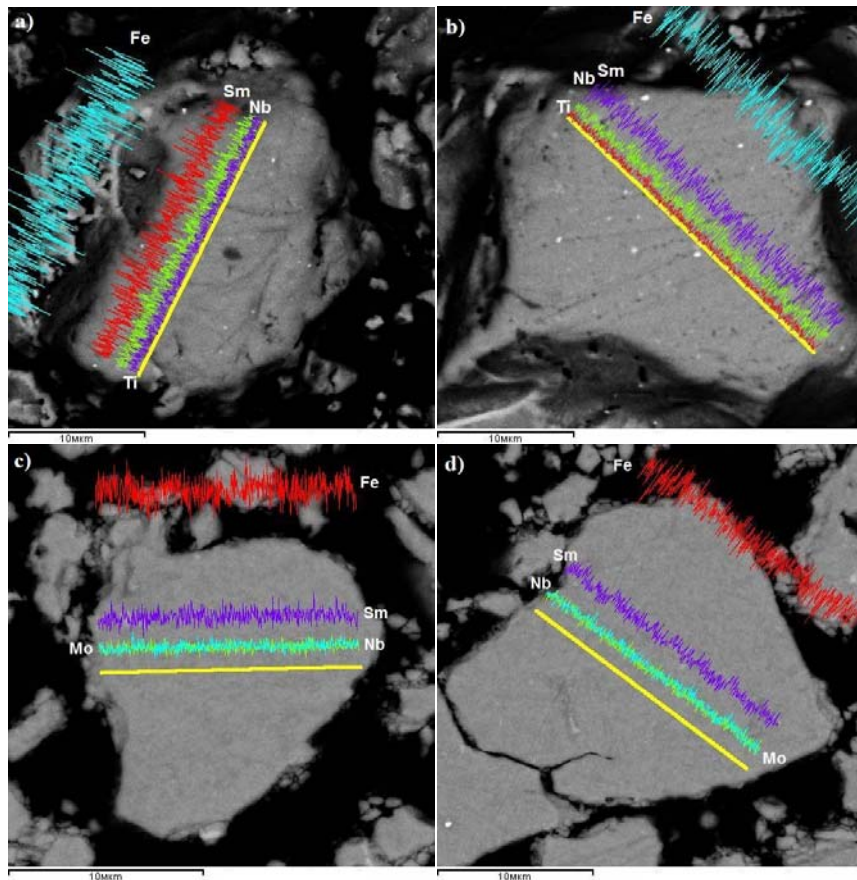


Figure-2. The distribution of elements in the volume of the powder particle: a) $\text{Sm}_2\text{Fe}_{15.7}\text{Nb}_{0.8}\text{Ti}_{0.5}$, b) $\text{Sm}_2\text{Fe}_{15.5}\text{NbTi}_{0.5}$, c) $\text{Sm}_2\text{Fe}_{15.7}\text{Nb}_{0.8}\text{Mo}_{0.5}$, d) $\text{Sm}_2\text{Fe}_{15.5}\text{NbMo}_{0.5}$.

The results of the X-ray phase analysis of the studied powders (Figure-3) have shown that the main

phase component in the sample is the phase with a crystal lattice corresponding to $\text{Th}_2\text{Zn}_{17}$ type with a small content



of α -Fe, samarium oxide, SmFe_3 and NbFe_2 . Formation of samarium oxide, probably, is caused by oxidation of samarium during preparation of the initial powder composition or in the process of mechanical alloying due to the use of insufficiently pure gas. Presence of α -Fe and SmFe_3 suggests that the chemical reaction of $\text{Sm}_2\text{Fe}_{17}$ formation was incomplete due to the formation of

intermetallic NbFe_2 . The addition of alloying elements results in a broadening and simultaneous shift of the peaks, belonging to $\text{Th}_2\text{Zn}_{17}$ -type crystalline lattice that is associated with the change in the crystalline lattice parameter when introducing alloying elements into the $\text{Th}_2\text{Zn}_{17}$ -type lattice (Table-1).

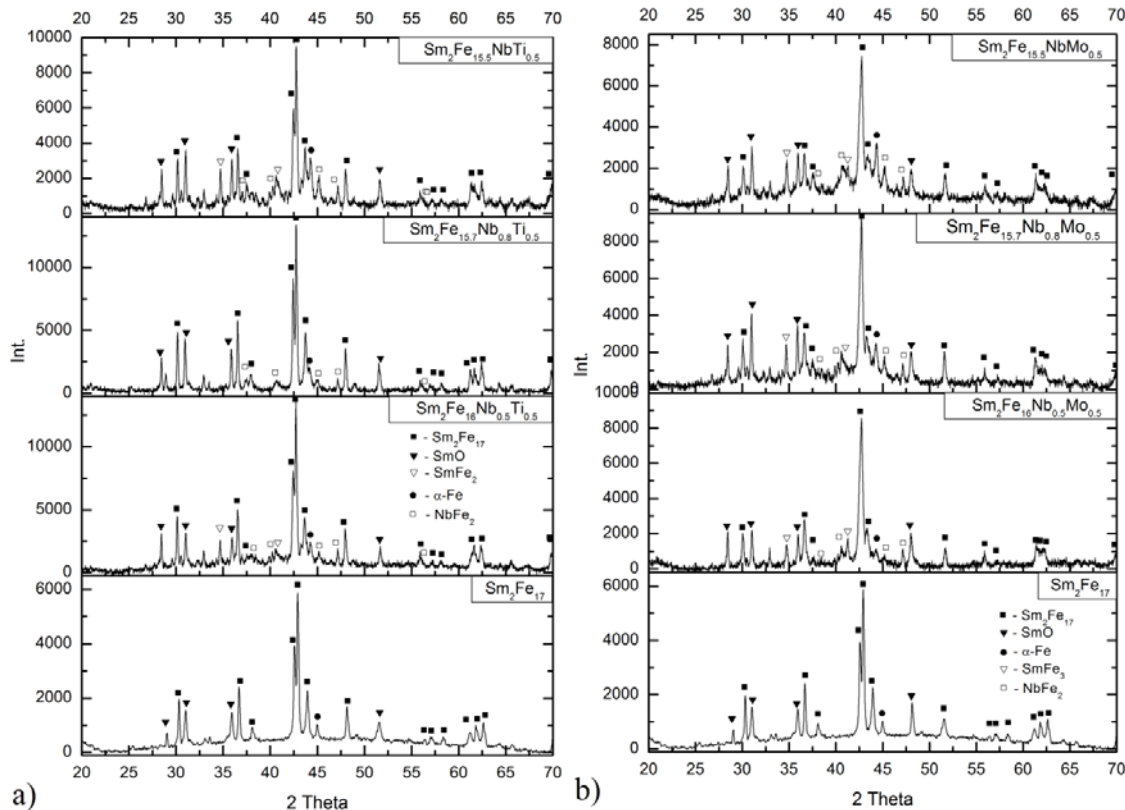


Figure-3. Changes in the phase composition of alloys: a) Sm-Fe-Nb-Ti, b) Sm-Fe-Nb-Mo.

Table-1. Changes in the $\text{Sm}_2\text{Fe}_{17}$ lattice parameter depending on alloying.

No.	Alloy	a, Å	c, Å	V, nm ³ /10 ³	c/a
1	$\text{Sm}_2\text{Fe}_{17}$	8.551	12.437	790.29	1.454
2	$\text{Sm}_2\text{Fe}_{16}\text{Nb}_{0.5}\text{Ti}_{0.5}$	8.5557	12.4922	791.92	1.460
3	$\text{Sm}_2\text{Fe}_{15.7}\text{Nb}_{0.8}\text{Ti}_{0.5}$	8.5535	12.4603	789.51	1.457
4	$\text{Sm}_2\text{Fe}_{15.5}\text{NbTi}_{0.5}$	8.5541	12.4784	790.76	1.459
5	$\text{Sm}_2\text{Fe}_{16}\text{Nb}_{0.5}\text{Mo}_{0.5}$	8.5267	12.5839	792.33	1.476
6	$\text{Sm}_2\text{Fe}_{15.7}\text{Nb}_{0.8}\text{Mo}_{0.5}$	8.5265	12.5817	792.17	1.476
7	$\text{Sm}_2\text{Fe}_{15.5}\text{NbMo}_{0.5}$	8.5355	12.5604	792.51	1.503

During the study of the Curie temperature it was revealed that the additional alloying of the initial $\text{Sm}_2\text{Fe}_{17}$ sample with titanium, molybdenum and niobium leads to an increase in the Curie temperature from 139 °C (initial alloy) up to 206 °C (Table-2). Determination of magnetic characteristics was carried out based on the parameters of a hysteresis loop (results of measurement are given in

Table-2). According to the results obtained, the samples contain the fraction with anisotropy field exceeding 2.2 T; thus the loops turned out to be asymmetric (Figure-4). The value of magnetization for the fore and reversal strokes does not match for the same reason. Introduction of Nb, Ti and Mo leads to a broadening of the hysteresis loop that means also increasing of residual magnetic induction and



coercive force. Hysteresis loop displacements both in vertical and in the horizontal direction can be caused by

presence of an antiferromagnetic phase due to there is exchange shift.

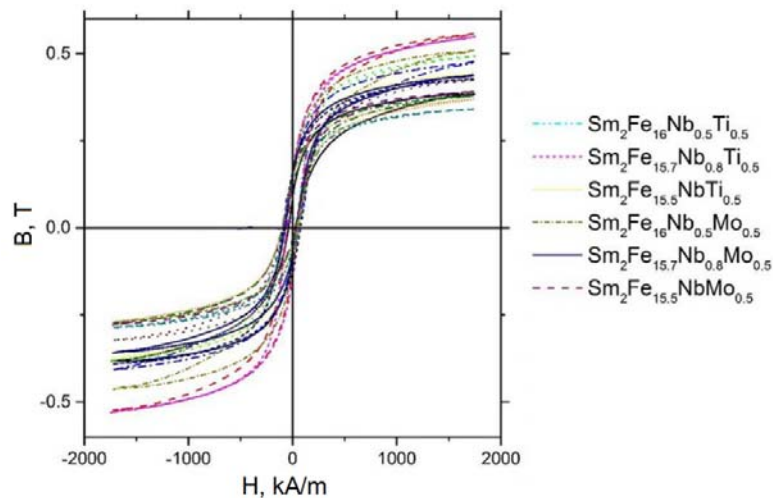


Figure-4. Hysteresis loops of Sm-Fe-Nb-Ti and Sm-Fe-Nb-Mo-based alloys obtained by mechanical alloying.

Table-2. Magnetic characteristics and Curie temperature of Sm-Fe-Nb-Ti and Sm-Fe-Nb-Mo-based alloys obtained by mechanical alloying

No.	Alloy	T _C , °C	H _c , kA/m	B _r , T	(BH) _{max} , kJ/m ³
1	Sm ₂ Fe ₁₇	139	28.1	0.09	0.862
2	Sm ₂ Fe ₁₆ Nb _{0.5} Ti _{0.5}	166.6	79.6	0.15	3.024
3	Sm ₂ Fe _{15.7} Nb _{0.8} Ti _{0.5}	149.3	62.8	0.16	2.840
4	Sm ₂ Fe _{15.5} NbTi _{0.5}	206.4	57.4	0.12	2.916
5	Sm ₂ Fe ₁₆ Nb _{0.5} Mo _{0.5}	187.5	68.5	0.14	3.091
6	Sm ₂ Fe _{15.7} Nb _{0.8} Mo _{0.5}	180	52.1	0.11	2.754
7	Sm ₂ Fe _{15.5} NbMo _{0.5}	184	38.7	0.09	3.852

4. CONCLUSIONS

The influence of complex alloying of the Sm₂Fe₁₇ with titanium, molybdenum and niobium on the Curie temperature (T_C) and magnetic properties were investigated. The alloying of Sm₂Fe₁₇ with titanium, molybdenum and niobium leads to an increase of the lattice parameter and volume of the unit cell without changing the lattice symmetry. It was revealed that complex alloying with titanium, molybdenum and niobium increases the Curie temperature from 139 °C (initial alloy) to 206 °C. By measurement the hysteretic properties of Sm₂Fe₁₇ based alloys obtained by mechanical alloying, it is revealed that alloying with titanium, molybdenum and niobium leads to a broadening of the hysteresis loops.

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