EFFECT OF NIOBIUM, TITANIUM AND MOLYBDENUM ADDITIONS TO Sm$_2$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 Sm$_2$Fe$_{17}$ alloy are presented. It is shown, that alloying of Sm$_2$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 Nd$_2$Fe$_{14}$B/α-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: Nd$_2$Fe$_{14}$B and the bottom limit - with spin-orientation phase transition Nd$_2$Fe$_{12}$B, which is observed at temperatures below -131 °C [10]. In ones of the first works for the alloying of Sm$_2$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 Sm$_{10,2}$Fe$_{55,8}$Nb$_{0,5}$ has the maximum Curie temperature. In the work [14] shown, that in Sm$_2$Fe$_{17-x}$ 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 Sm$_2$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 Sm$_2$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 Sm$_2$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$_2$-Fe-Nb-Ti and Sm$_2$-Fe-Nb-Mo-based alloys of various compositions (Sm$_2$Fe$_{16}$Nb$_{0,5}$Ti$_{0,5}$, Sm$_2$Fe$_{15,5}$Nb$_{0,8}$Ti$_{0,5}$, Sm$_2$Fe$_{15,5}$NbTi$_{0,5}$, Sm$_2$Fe$_{14}$Nb$_{0,5}$Mo$_{0,5}$, Sm$_2$Fe$_{15,5}$Nb$_{0,8}$Mo$_{0,5}$, Sm$_2$Fe$_{14,3}$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 ($d_{90} < 300$ μm), samarium ($d_{90} < 700$ μm), 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 Brucker D8 Advance in CuKα-radiation ($\lambda = 1,5418$Å). 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 (± 4 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 $d_{50} = 10 \mu m$ (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).

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 Th$_2$Zn$_{17}$ type with a small content
of $\alpha$-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 $\alpha$-Fe and SmFe$_3$ suggests that the chemical reaction of Sm$_2$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 Th$_2$Zn$_{17}$-type crystalline lattice that is associated with the change in the crystalline lattice parameter when introducing alloying elements into the Th$_2$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.](image)

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

During the study of the Curie temperature it was revealed that the additional alloying of the initial Sm$_2$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

<table>
<thead>
<tr>
<th>No.</th>
<th>Alloy</th>
<th>$T_C$, °C</th>
<th>$H_c$, kA/m</th>
<th>$B_r$, T</th>
<th>$(BH)_{max}$, kJ/m³</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Sm$<em>2$Fe$</em>{17}$</td>
<td>139</td>
<td>28.1</td>
<td>0.09</td>
<td>0.862</td>
</tr>
<tr>
<td>2</td>
<td>Sm$<em>2$Fe$</em>{16}$Nb$<em>{0.5}$Ti$</em>{0.5}$</td>
<td>166.6</td>
<td>79.6</td>
<td>0.15</td>
<td>3.024</td>
</tr>
<tr>
<td>3</td>
<td>Sm$<em>2$Fe$</em>{15.7}$Nb$<em>{0.8}$Ti$</em>{0.5}$</td>
<td>149.3</td>
<td>62.8</td>
<td>0.16</td>
<td>2.840</td>
</tr>
<tr>
<td>4</td>
<td>Sm$<em>2$Fe$</em>{14.5}$NbTi$_{0.5}$</td>
<td>206.4</td>
<td>57.4</td>
<td>0.12</td>
<td>2.916</td>
</tr>
<tr>
<td>5</td>
<td>Sm$<em>2$Fe$</em>{16}$Nb$<em>{0.5}$Mo$</em>{0.5}$</td>
<td>187.5</td>
<td>68.5</td>
<td>0.14</td>
<td>3.091</td>
</tr>
<tr>
<td>6</td>
<td>Sm$<em>2$Fe$</em>{15.7}$Nb$<em>{0.8}$Mo$</em>{0.5}$</td>
<td>180</td>
<td>52.1</td>
<td>0.11</td>
<td>2.754</td>
</tr>
<tr>
<td>7</td>
<td>Sm$<em>2$Fe$</em>{15.5}$NbMo$_{0.5}$</td>
<td>184</td>
<td>38.7</td>
<td>0.09</td>
<td>3.852</td>
</tr>
</tbody>
</table>

4. CONCLUSIONS

The influence of complex alloying of the Sm$_2$Fe$_{17}$ with titanium, molybdenum and niobium on the Curie temperature ($T_C$) and magnetic properties were investigated. The alloying of Sm$_2$Fe$_{17}$ 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 Sm$_2$Fe$_{17}$ 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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REFERENCES


