

The Influence of Aluminium Addition on the Structure and Magnetic Properties of the Pseudobinary $\text{Sm}_2(\text{Fe}_{1-x}\text{Al}_x)_{17}$ Alloys and Their Hydrides

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ABSTRACT: Results of XRD analysis and magnetic measurements performed on $\text{Sm}_2(\text{Fe}_{1-x}\text{Al}_x)_{17}$ ($x=0.1-0.4$) samples and their hydrides are reported. X-ray powder diffraction studies using the Rietveld method have shown that aluminium alloys crystallize in the $\text{Th}_2\text{Zn}_{17}$ type rhombohedral structure and synthesized hydrides preserved the same type structure as their parent compounds. It was established that the replacement of Al for Fe in $\text{Sm}_2\text{Fe}_{17}$ causes a monotonic increase in the structural parameters for $\text{Sm}_2(\text{Fe}_{1-x}\text{Al}_x)_{17}$ compounds. The specific saturation magnetization (σ_s), specific remanent magnetization (σ_r) and specific coercive force by magnetization (jH_c) of all samples carried out at room temperature ($H=9$ T) decreases by Al substitution. As well as the magnetic properties of hydrides with analogous tendency has been studied. The $\text{Sm}_2(\text{Fe}_{1-x}\text{Al}_x)_{17}\text{H}_y$ compounds were prepared by heat-treating powders of corresponding two-phases initial alloys in H_2 gas. The introduction of hydrogen atoms leads to an increase in the lattice constants and unit cell volume, but the increase becomes less noticeably with raising Al concentration.

KEY WORDS: rare earth compounds; hydrides based on Sm-Fe-Al alloys; crystal structure; X-ray phase analysis; electron probe microanalysis; magnetic measurements

ВЛИЯНИЕ АЛЮМИНИЯ НА СТРУКТУРУ И МАГНИТНЫЕ СВОЙСТВА ПСЕВДОБИНАРНЫХ СПЛАВОВ $\text{Sm}_2(\text{Fe}_{1-x}\text{Al}_x)_{17}$ И ИХ ГИДРИДОВ

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РЕЗЮМЕ: Сообщается о результатах РФА и магнитных измерений, проведенных на $\text{Sm}_2(\text{Fe}_{1-x}\text{Al}_x)_{17}$ ($x=0.1-0.4$) образцах и их гидридах. Рентгенографические исследования порошковых образцов с применением метода Ритвелда показали, алюминиевые сплавы кристаллизуются в ромбоэдрическом структурном типе $\text{Th}_2\text{Zn}_{17}$, синтезированные гидриды сохранили тот же тип структуры, что и родственные соединения. Установлено, замещение железа алюминием в $\text{Sm}_2\text{Fe}_{17}$ приводит к монотонному увеличению структурных параметров $\text{Sm}_2(\text{Fe}_{1-x}\text{Al}_x)_{17}$. Удельная намагниченность насыщения, остаточная удельная намагниченность насыщения и остаточная коэрцитивная сила по намагниченности всех образцов, измеренные при комнатной температуре в магнитных полях до 9 Тл, убывают по мере замещения алюминием. Также были проведены измерения магнитных свойств гидридов с аналогично выявленной

тенденцией. Соединения $\text{Sm}_2(\text{Fe}_{1-x}\text{Al}_x)_{17}\text{H}_y$ получены термообработкой порошков соответствующих исходных двухфазных образцов в атмосфере водорода. Внедрение атомов водорода является причиной увеличения периодов и объема элементарной ячейки, что становится менее заметным с ростом концентрации алюминия.

КЛЮЧЕВЫЕ СЛОВА: редкоземельные соединения; гидриды на основе сплавов Sm-Fe-Al; кристаллическая структура; РФА; электронно-зондовый микроанализ; магнитные измерения

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1 Introduction

It is known that substitutional and interstitial impurities (atoms of metals) usually modify hard-magnet properties of Sm-Fe materials due to changes in hybridization produced by changes in bonding and volume. In particular the magnetic properties of the pseudobinary $\text{Sm}_2\text{Fe}_{17-x}\text{T}_x$ and $\text{Sm}_{2-y}\text{M}_y\text{Fe}_{17}$ (T – transition metal, M – rare earth) compounds with substituted iron or samarium for other elements in the $\text{Sm}_2\text{Fe}_{17}$ structure may be modified remarkably [1-4]. Thus such fundamental intrinsic magnetic characteristics of permanent magnets as Curie temperature, magneto crystalline anisotropy are transformed. In order to achieve high coercivities it is necessary to receive larger values of the uniaxial magnetocrystalline anisotropy. The ternary $\text{Sm}_2\text{Fe}_{17-x}\text{Al}_x$ compounds and nitrides based on them have been studied extensively [5-7]. It was found that T_c of the parent compounds and nitrides depends on the Al concentration (the increase in Curie temperature with Al has been attributed to the expansion of the Fe-Fe bonds) and decreases with raising x_{Al} . In spite of the fact that for the Sm-Fe-H system has been not observed a change of the anisotropy from planar to uniaxial unlike $\text{Sm}_2\text{Fe}_{17}\text{X}_k$ (X = C or N) it is worth noting the effect substitutional-interstitial modification on the electronic structure, exchange interactions, magnetic phase transitions occurring in the $\text{Sm}_2\text{Fe}_{17}$ compound.

The present paper we have investigated the structural and some magnetic features of non single-phase Sm-Fe-Al alloys and their hydrides, in order to reveal the effect of partially replacement of iron for Al on the fundamental magnetic properties and structure of $\text{Sm}_2\text{Fe}_{17-x}\text{Al}_x\text{H}_z$ -based pseudo quadruples compounds. Although the results on the stoichiometric $\text{Sm}_2\text{Fe}_{17-x}\text{Al}_x$ compounds are reported in the literature are systematically, which are observed no considerable diffraction from impurity phase, we report the results of our investigation on non single-single samples and give here some additional information on hydrides $\text{Sm}_2\text{Fe}_{17-x}\text{Al}_x\text{H}_z$ in addition to presented results earlier by other authors [8].

2 Experimental procedure

The samples having nominal compositions $\text{Sm}_2(\text{Fe}_{1-x}\text{Al}_x)_{17}$ were prepared by melting the constituent elements in an arc furnace ($x=0.1-0.3$) and induction melting method ($x=0.4$) under purified argon atmosphere, followed by annealing in vacuum at 1000°C for 41 hours and subsequently cooling to room temperature. The purities of the component metals were at least 99 wt. %. The first three ingots were remelted several times to insure the microstructural homogeneity using a non consumable tungsten electrode and a water-cooled copper tray. Titanium was used as a getter during arc-melting. In order to compensate for the Sm evaporation losses during melting was added an appropriate excess (5 wt. % Sm).

The structure, phase purity and chemical composition of the alloys were checked using standard X-ray diffractometry, electron microprobe analysis and semi-quantitative analysis by X-ray fluorescence. The phase of the alloy powders and structural parameters of the parent compounds and corresponding hydrides was examined by X-ray diffraction analysis with Co K_α radiation at 40kV and 30mA. The X-ray data were obtained on a diffractometer DRON-4-07 with scans 2θ ranging from 20° to 120°, steps 0.05° and counting times of 5 s per point, both before and after hydrogenation. The refinement of diffraction patterns was analyzed via Rietveld refinement by Rietan-program for multiphase materials. The surface of the samples was studied by scanning electron microscopy on a LEO EVO50 XVP instrument (Karl Zeiss, Germany) at an accelerating voltage of 25 kV. The composition was determined using energy dispersive microanalysis on an INCA energy 350 detector (Oxford Instruments, England) at a voltage of 15 kV. X-Ray Fluorescence Spectrometer ZSX Primus II (Rigaku, Japan)

was employed to perform the elemental composition. The results were not in good agreement with the nominal compositions.

Alloy samples having a mass approximately 10 g were placed in the stainless steel autoclaves in the special testing devices, which was evacuated for about 1 hour at room temperature. Then alloy were allowed to react with hydrogen gas under pressure. Hydrogenation was performed by heat treating pulverized $\text{Sm}_2(\text{Fe}_{1-x}\text{Al}_x)_{17}$ in purified H_2 gas (99.999% purity) at a pressure up to 30 atm at $200^\circ\text{C}\pm 10^\circ\text{C}$ until pressure dropped finally and it leads to formation of stable hydrides. The reaction container was cooled to room temperature. The hydrogen content was determined by means of the Van der Waals equation.

The specific saturation magnetization (σ_s), specific remanent magnetization (σ_r) and specific coercive force by magnetization (jH_c) of the synthesized materials was measured at room temperature with the external magnetic field of 9 T using a PPMS EverCool-II magnetometer. The values of σ_s and σ_r are given in emu per kilogramme or amp meters squared per kilogramme.

3 Results and discussion

3.1 Characterization of $\text{Sm}_2(\text{Fe}_{1-x}\text{Al}_x)_{17}$ parent alloys: the structure, phase purity and chemical composition

Both, microprobe and X-ray powder diffraction analysis indicate that the ternary $\text{Sm}_2(\text{Fe}_{1-x}\text{Al}_x)_{17}$ samples contain in addition to the main phase a considerable amount (≤ 35 wt.%) of α -Fe(Al) as second phase. The results of the investigations on the phase composition of the homogenised samples by SEM-EDX and XRD are listed in Table 1. The XRD analysis reveals that the dominant phase $\text{Sm}_2(\text{Fe}, \text{Al})_{17}$ has a rhombohedral structure of $\text{Th}_2\text{Zn}_{17}$ -type (space group $R\bar{3}m$). In other words the replacement of Al for Fe does not change the structure regarding $\text{Sm}_2\text{Fe}_{17}$ compound. It is clearly seen that only one of the four compositions is observed, $\text{Sm}_{1.98}\text{Fe}_{9.84}\text{Al}_{7.1}$ in relative agreement with the expected stoichiometry. It can be assumed that this phenomenon is associated with a more homogeneous melt of the constituent metals during the induction melting method.

Table 1 Annealing conditions, chemical and structural analysis of the compounds

Nominal composition	Annealing (time/temperature)	Chemical composition	Structural type	Phase amount (wt.%)
$\text{Sm}_2\text{Fe}_{15.3}\text{Al}_{1.7}$	41h/1000 °C	$\text{Sm}_2\text{Fe}_{14.9}\text{Al}_{2.1}$	$\text{Th}_2\text{Zn}_{17}$ ($R\bar{3}m$)	57
		α -Fe(Al)	W ($Im\bar{3}m$)	43
$\text{Sm}_2\text{Fe}_{13.6}\text{Al}_{3.4}$	41h/1000 °C	$\text{Sm}_2\text{Fe}_{13.2}\text{Al}_{3.8}$	$\text{Th}_2\text{Zn}_{17}$ ($R\bar{3}m$)	72
		α -Fe(Al)	W ($Im\bar{3}m$)	28
$\text{Sm}_2\text{Fe}_{11.9}\text{Al}_{5.1}$	41h/1000 °C	$\text{Sm}_2\text{Fe}_{10.7}\text{Al}_{6.3}$	$\text{Th}_2\text{Zn}_{17}$ ($R\bar{3}m$)	75
		α -Fe(Al)	W ($Im\bar{3}m$)	25
$\text{Sm}_2\text{Fe}_{10.2}\text{Al}_{6.8}$	41h/1000 °C	$\text{Sm}_2\text{Fe}_{9.9}\text{Al}_{7.1}$	$\text{Th}_2\text{Zn}_{17}$ ($R\bar{3}m$)	96
		α -Fe(Al)	W ($Im\bar{3}m$)	4

The amount of unreacted α -Fe and Sm-rich phase which are contained in alloys before homogenisation procedure can be reduced significantly by a time consuming homogenization treatment. The homogenization was carried out at a temperature above the melting points of the Sm-rich phases, all observed phases were detected by SEM-EDX after this treatment. The typical microstructure obtained after annealing of alloys with nominal compositions ' $\text{Sm}_2\text{Fe}_{15.3}\text{Al}_{1.7}$ ', ' $\text{Sm}_2\text{Fe}_{10.2}\text{Al}_{6.8}$ ', ' $\text{Sm}_2\text{Fe}_{11.9}\text{Al}_{5.1}$ ' and ' $\text{Sm}_2\text{Fe}_{10.2}\text{Al}_{6.8}$ ' are presented in Fig. 1. As can be seen also from this figure, there are some white secretions as surface defects were formed in the time of the polishing test samples.

Although it was assumed that the partial replacement of Fe by Al and some Sm excess will lead to the essential suppression of α -Fe, in fact this is not so. In respect to the phase composition of this alloys, in each of the three materials arc melted a significant content of the impurity soft magnetic α -Fe(Al) phase was observed, which is shown in images by dark color. By reason of the samarium shortage in the main phase of these materials were received such samples with excess of the phase based on α -Fe. Obviously, in order to have pseudobinary aluminium compounds without any amount of α -Fe by this method is need to take samarium more to compensate for its loss. The $\text{Sm}_2\text{Fe}_{11.9}\text{Al}_{5.1}$ sample is characterized by a strongly heterogeneous dendrite-like structure, which could arise from the non-crystallized metal in the as-cast sample (Fig. 1(c)).

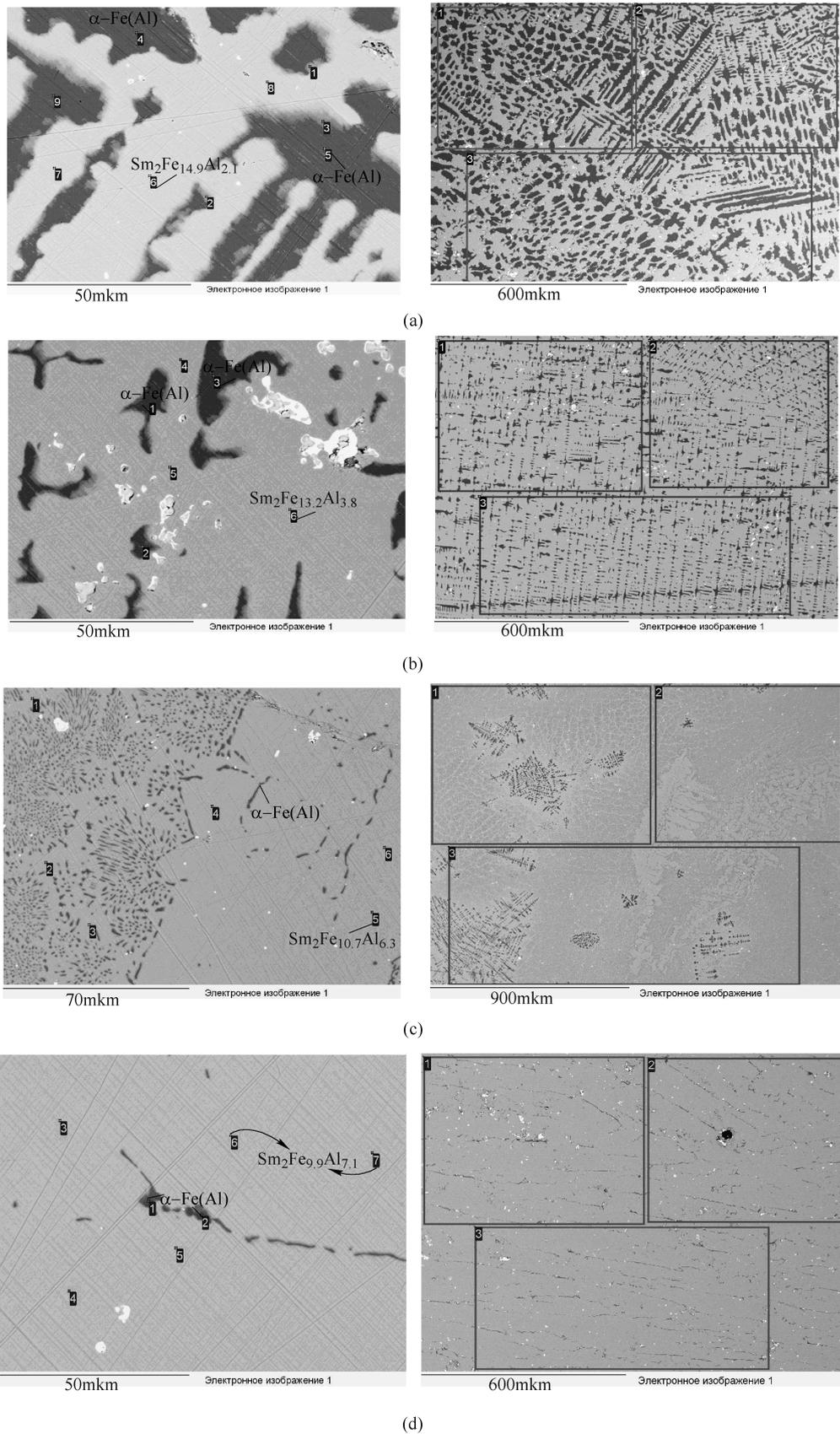


Fig.1 SEM micrographs of $\text{Sm}_2(\text{Fe}_{1-x}\text{Al}_x)_{17}$ ($x = 0.1, 0.2, 0.3, 0.4$) alloys after annealing at 1000°C for 41 hours: (a) SEM image of the ' $\text{Sm}_2\text{Fe}_{15.3}\text{Al}_{1.7}$ ' sample, (b) SEM picture of the ' $\text{Sm}_2\text{Fe}_{13.6}\text{Al}_{3.4}$ ' sample, (c) SEM image of the ' $\text{Sm}_2\text{Fe}_{11.9}\text{Al}_{5.1}$ ' sample, (d) SEM picture of the ' $\text{Sm}_2\text{Fe}_{10.2}\text{Al}_{6.8}$ ' sample (Here the dark phase is a pure $\alpha\text{-Fe(Al)}$ and grey phase has the $\text{Sm}_2(\text{Fe, Al})_{17}$ composition)

Typical X-ray diffraction patterns of the ‘Sm₂Fe_{15.3}Al_{1.7}’, ‘Sm₂Fe_{10.2}Al_{6.8}’, ‘Sm₂Fe_{11.9}Al_{5.1}’ and ‘Sm₂Fe_{10.2}Al_{6.8}’ alloys are illustrated in Fig. 2 respectively. Indices of the major peaks for the rhombohedral Th₂Zn₁₇ phase are indicated in this illustration. Lattice parameters and unit cell volume have been determined by Rietveld refinement and are given in Table 2. From these results, in all the Al substituted Sm₂(Fe_{1-x}Al_x)₁₇ samples the unit cell volume increases linearly with raising Al content.

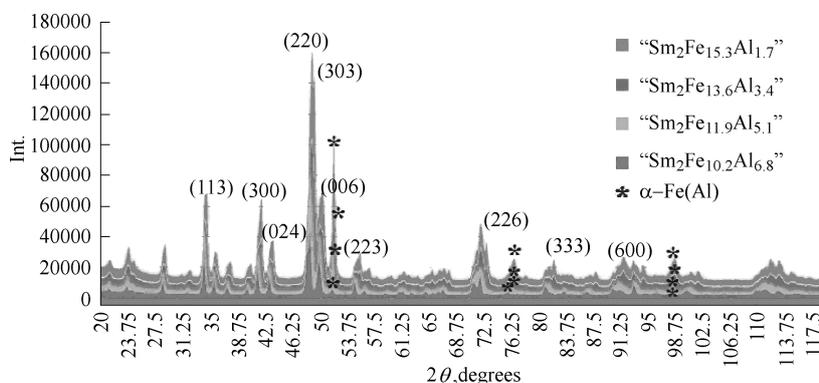


Fig.2 X-ray powder patterns of Sm₂(Fe,Al)₁₇ alloys: (1) ‘Sm₂Fe_{15.3}Al_{1.7}’, (2) ‘Sm₂Fe_{13.6}Al_{3.4}’, (3) ‘Sm₂Fe_{11.9}Al_{5.1}’, (4) ‘Sm₂Fe_{10.2}Al_{6.8}’ (Intensity are given in the relative units)

3.2 Hydrogen interaction with Sm₂(Fe, Al)₁₇ and characterization of Sm-Fe-Al hydrides

It was revealed that from Sm₂(Fe_{1-x}Al_x)₁₇ has been formed the corresponding Sm₂(Fe_{1-x}Al_x)₁₇H_y by gas-phase reaction. As it can see from Fig. 3 that the diffraction peaks of hydrides are shifted to lower angles except of the α-Fe(Al) peak and the intensity ratios of the diffractions are approximately the same before and after hydrogenation. The X-ray reflections of hydrides are shifted to lower angles as a result of the lattice expansion compared with starting samples. In both cases, the cell volumes expand by the substituting Fe by bigger size atoms Al and introducing H atoms. However, according to X-ray diffraction analysis all the Sm₂(Fe_{1-x}Al_x)₁₇H_y hydrides remain in the 2 : 17 structure of the original compounds and the hydrogen atoms only enter into interstitial sites. Table 2 summaries the parameters obtained from the diffraction studies of these materials.

Table 2 Crystallographic data for the Sm₂(Fe,Al)₁₇ alloys and Sm₂(Fe,Al)₁₇H_y hydrides (Th₂Zn₁₇-type; sp. gr. $R\bar{3}m$; no. 166) from the Rietveld refinements of the XRD data

Sample	<i>a</i> /Å	<i>c</i> /Å	<i>V</i> /Å ³	$\Delta a/a_0$ %	$\Delta c/c_0$ %	$\Delta V/V_0$ %
Sm ₂ Fe _{14.9} Al _{2.1} H _{3.6}	8.698(1)	12.655(3)	827.3	1.1	1.1	~3
Sm ₂ Fe _{14.9} Al _{2.1}	8.603(4)	12.522(3)	802.7			
Sm ₂ Fe _{13.3} Al _{3.7} H _{2.1}	8.700(2)	12.684(9)	831.6	0.7	0.8	2
Sm ₂ Fe _{13.3} Al _{3.7}	8.643(7)	12.596(3)	815.6			
Sm ₂ Fe _{10.7} Al _{6.3} H ₂	8.750(2)	12.763(2)	846.1	0.6	0.7	1.9
Sm ₂ Fe _{10.7} Al _{6.3}	8.695(3)	12.670(5)	829.9			
Sm ₂ Fe _{9.9} Al _{7.1} H _{1.8}	8.799(3)	12.813(6)	859.2	0.5	0.6	1.5
Sm ₂ Fe _{9.9} Al _{7.1}	8.759(0)	12.740(4)	846.5			

In addition, the hydrogenation brings some expansion of the unit cell: the value of $\Delta V/V_0$ changes from 1.5% to 3% with respect to the parent alloys. It is worth noting that $(\Delta V/V)/H$ and the lattice parameters *a* and *c* of Sm₂(Fe_{1-x}Al_x)₁₇H_y monotonously decreases with Al content. Consequently, the expansion effect of hydrogen is reduced due to the size effect of aluminium replacement.

3.3 Magnetic properties of Sm₂(Fe_{1-x}Al_x)₁₇ parent alloys and their hydrides

Fig. 4 shows the magnetization curves of Sm₂(Fe_{1-x}Al_x)₁₇ alloys (a)-(d) and Sm₂(Fe_{1-x}Al_x)₁₇H_y hydrides (e)-(h)

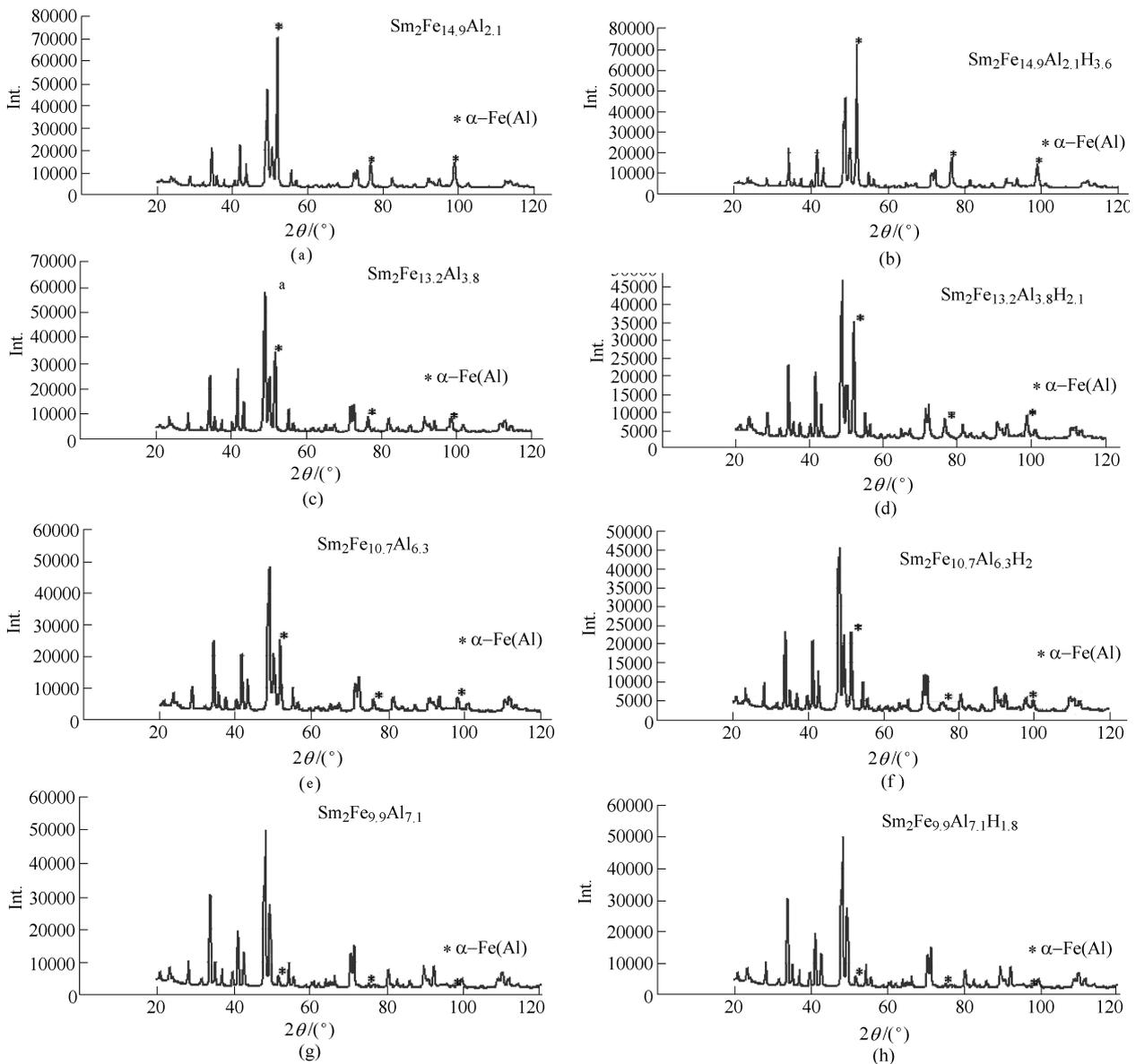


Fig.3 X-ray diffraction patterns for $\text{Sm}_2(\text{Fe}_{1-x}\text{Al}_x)_{17}$ alloys (a, c, e, g) and their $\text{Sm}_2(\text{Fe}_{1-x}\text{Al}_x)_{17}\text{H}_y$ hydrides (b, d, f, h). Intensity are given in the relative units. The symbol * means $\alpha\text{-Fe(Al)}$ phase, the other lines belong to phase of type 2:17 or its hydride

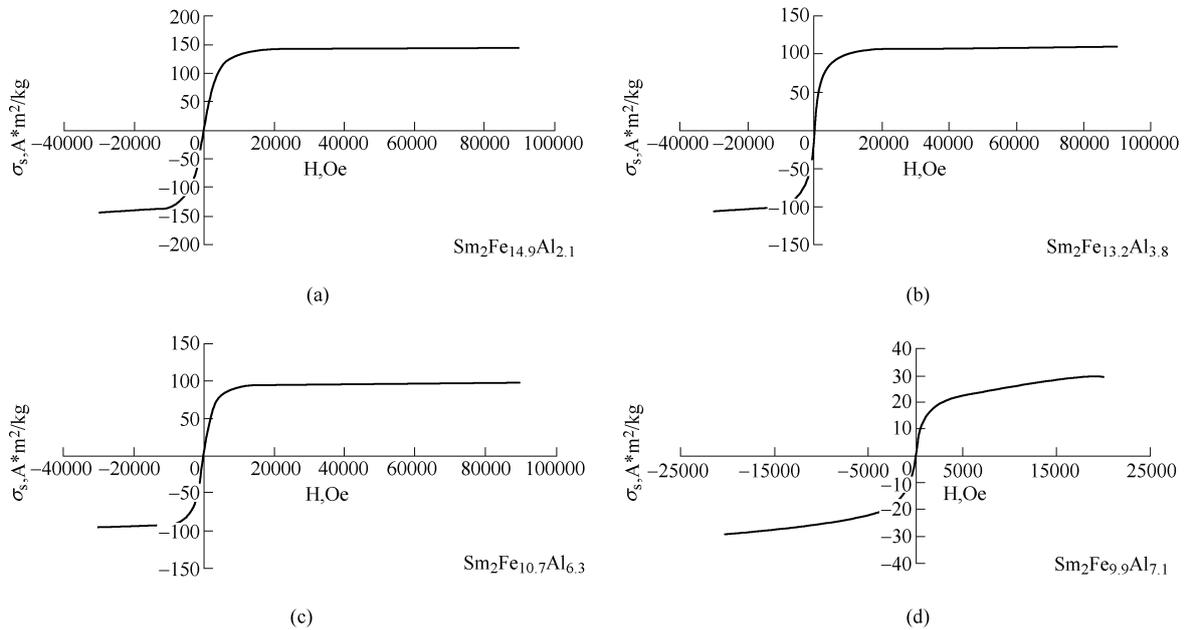
measured at room temperature. It is found that the saturation magnetization in a maximum field of 9 T at room temperature of both hydrides and the parent compounds decreases monotonic with increasing Al concentration (Fig. 5). It should be noted that this decrease is much faster than in the case of a simple magnetic dilution. Comparing Fig. 4 (a)-(d) with (e)-(h), as can be seen that the introduction of hydrogen leads to an increase in the saturation magnetization, but the amplitude of the increase decreases with Al concentration. Besides, it can be seen from curves of Fig. 4 specific coercive force by magnetization becomes smaller with increasing Al content. From these observed structural and magnetic measurements, this can be related to the fact that the substitution of Al for Fe in generally hinders the introduction of hydrogen atoms.

The above-mentioned heterogeneity of the starting alloy with a nominal composition ‘ $\text{Sm}_2\text{Fe}_{11.9}\text{Al}_{5.1}$ ’ has affected its magnetic characteristics as a result of which in Figs. 4(c) and 5 we observe some deviation of the value σ_s from the trend line.

4 Conclusions

From the above experimental results, we pointed the following conclusions. Pseudobinary $\text{Sm}_2(\text{Fe}_{1-x}\text{Al}_x)_{17}$

The hysteresis loops of the initial $\text{Sm}_2\text{Fe}_{17-x}\text{Al}_x$ alloys



The hysteresis loops of the $\text{Sm}_2\text{Fe}_{17-x}\text{Al}_x\text{H}_y$ hydrides

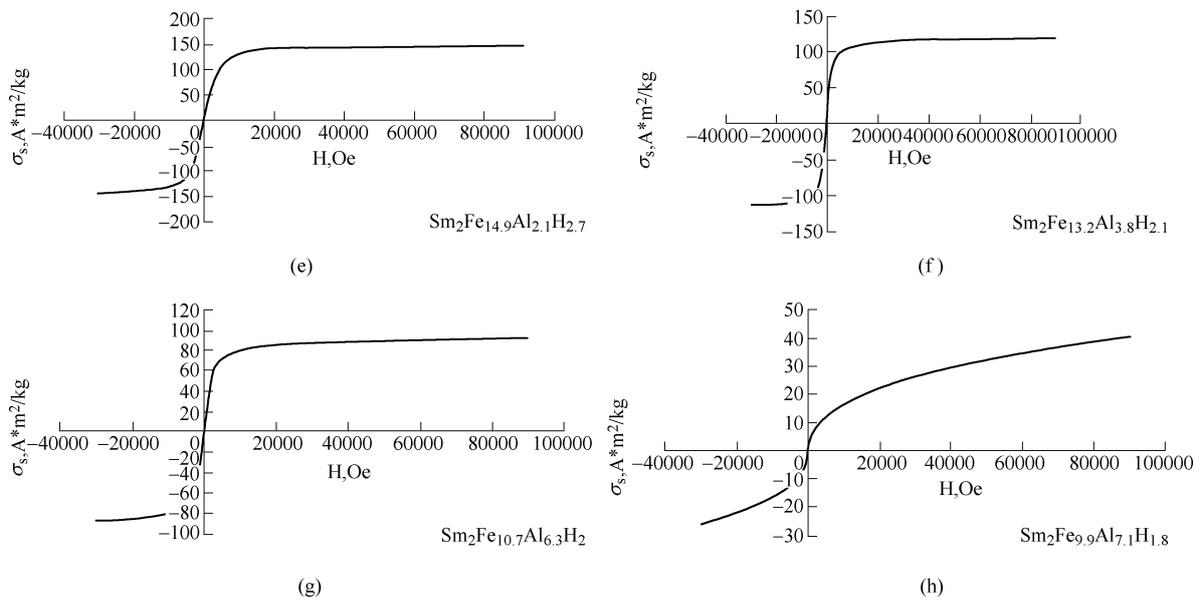


Fig.4 The hysteresis loops of the initial $\text{Sm}_2\text{Fe}_{17-x}\text{Al}_x$ alloys (a)-(d) and $\text{Sm}_2\text{Fe}_{17-x}\text{Al}_x\text{H}_y$ hydrides (e)-(h) measured at room temperature with the external magnetic field up to 9 T

samples with x up to 0.4 have been studied here and are shown to be non-single phased materials where the dominant phase $\text{Sm}_2(\text{Fe}, \text{Al})_{17}$ has a rhombohedral structure of $\text{Th}_2\text{Zn}_{17}$ -type (space group $R\bar{3}m$). All investigated hydrides have the same structure as the parent materials. Substitution of iron by aluminium in $\text{Sm}_2\text{Fe}_{17}$ leads to a volume increase and the introduction of hydrogen results in a further increase in lattice constants, unit-cell volume. However the content of the absorbed hydrogen amount and $\Delta V/V$ decrease ($\Delta V/V$ is

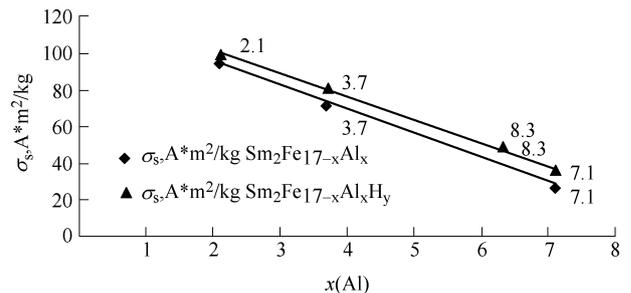


Fig.5 The saturation magnetization σ_s as a function of Al concentration x for $\text{Sm}_2\text{Fe}_{17-x}\text{Al}_x$ and $\text{Sm}_2\text{Fe}_{17-x}\text{Al}_x\text{H}_y$ samples

from 3% to 1.5 %) with increasing Al in hydrides. It was established that as the concentration of Al increases in both hydrides and the parent alloys, the specific saturation magnetization σ_s decreases monotonously. The values of σ_s of $\text{Sm}_2(\text{Fe}_{1-x}\text{Al}_x)_{17}$ and their corresponding hydrides are below values than these of $\text{Sm}_2\text{Fe}_{17}$ and $\text{Sm}_2\text{Fe}_{17}\text{H}_z$, respectively.

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