The influence of Fe content on phase-structural state of Sm_2Co_{17} compound during hydrogen-vacuum treatment

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Introduction

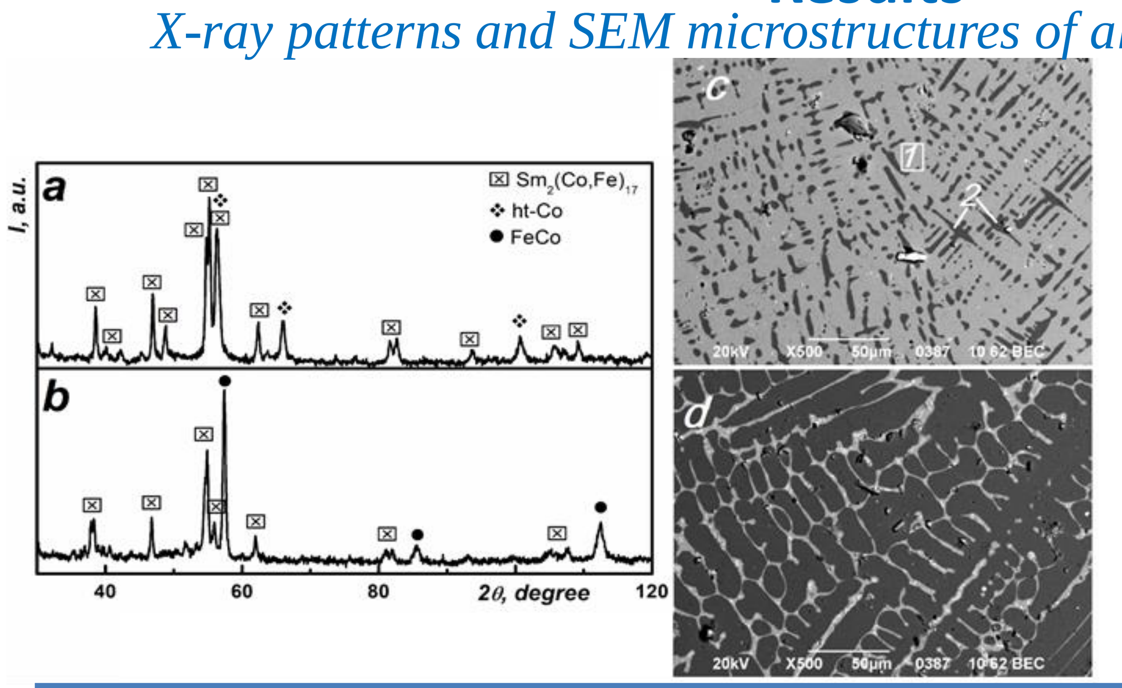
Rare-earth permanent magnets based on the $Nd_2Fe_{14}B$, $SmCo_5$, and Sm_2Co_{17} compounds have the highest coercivity, remanence, and maximum energy product among all known materials [1]. According to the theoretical prediction, the formation of the nanostructured state in REM magnets-nanocomposites will significantly increase their magnetic properties as the result of creation of the mechanism of the exchange interaction [2]. At the same time, it is important that the magnetic material must be two-phase and consists of both a magnetically hard phase with high saturation magnetization and a magnetically soft phase with high coercive force, which allows to almost double the value of the specific magnetic energy [3]. One of the promising methods for forming such a phase composition and microstructure is the treatment of hydride-forming materials in hydrogen [4] using hydrogenation, disproportionation, desorption, and recombination (HDDR) [5]. One of the usual methods of forming a two-phase state in ferromagnetic alloys of the Sm-Co system is doping with iron, which significantly affects the magnetic properties, the nature of the change of which is different for the $SmCo_5$ and Sm_2Co_{17} compounds [6]. At the same time, most publications on the formation of the nanostructured state in Sm-Co alloys describe $SmCo_5$/α-Fe nanocomposites. The possibility of HDDR implementation for the nanostructuring of Sm_2Co_{17}-based alloys, as well as the iron influence on phase transformations in this compound during hydrogen treatment, is insufficiently covered in the literature. However, this compound possesses higher coercive force, corrosion resistance, and lower temperature coefficient and is used for permanent magnets in high tech fields like aerospace and the military industry [7]. The purpose of this work is to study the influence of iron content in alloys based on the Sm_2Co_{17} compound on the features of the microstructure and phase transformations during hydrogen-vacuum treatment by the HDDR method.

Methods

Samples of $Sm_2Co_{17-x}Fe_x$ alloys (x = 2, 4, 6 and 8) were synthesised by melting the initial components with a purity of at least 99.9 % in an electric arc furnace under refined argon. Features of HDDR in the investigated alloys were studied by differential thermal analysis (DTA) during heating under hydrogen, and by the measuring of the hydrogen pressure in the chamber during heating disproportionated products in a vacuum [8]. The heating rate was 5 °C/min, while cooling was carried out without speed control. Disproportionation of the alloys was realized under the initial hydrogen pressure of 0.5; 2.0 and 4.0 MPa, the maximum heating temperature during heating in hydrogen and vacuum was 950 °C. X-ray diffraction (XRD) analysis of the studied materials was caried out using DRON-3M diffractometer with Fe-Kα radiation. X-ray patterns were identified using the PowderCell [9] and FullProf [10] software packages. The microstructure of the alloys was observed by an EVO-40XVP electron scanning microscope equipped by energy-dispersive X-ray spectrometer INCA ENERGY 350 for analysis of element composition. A mixture of nitrogen acid (2.5 and 5 vol. %) and ethyl alcohol was used for etching. The materials were investigated in the polished and etched states.

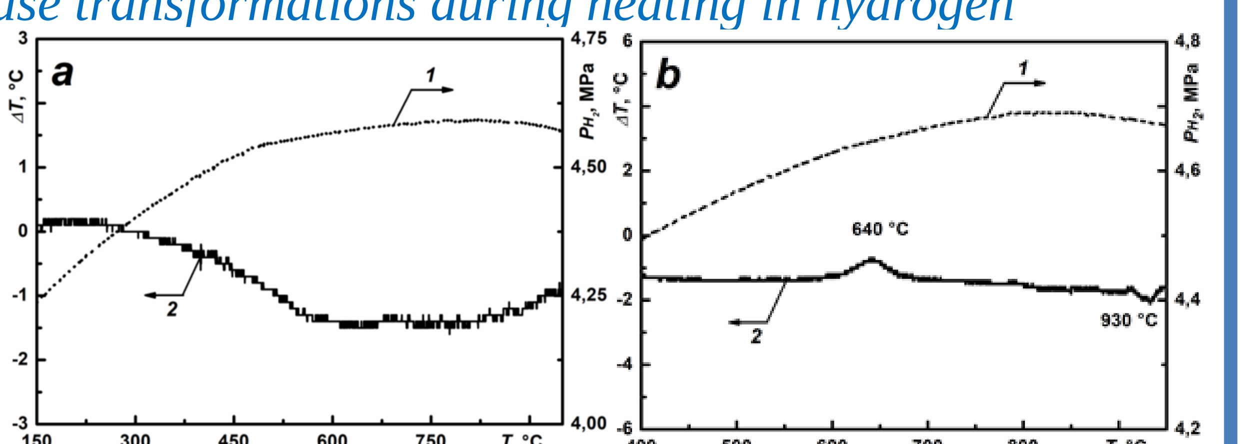
Results

X-ray patterns and SEM microstructures of alloys in the initial state



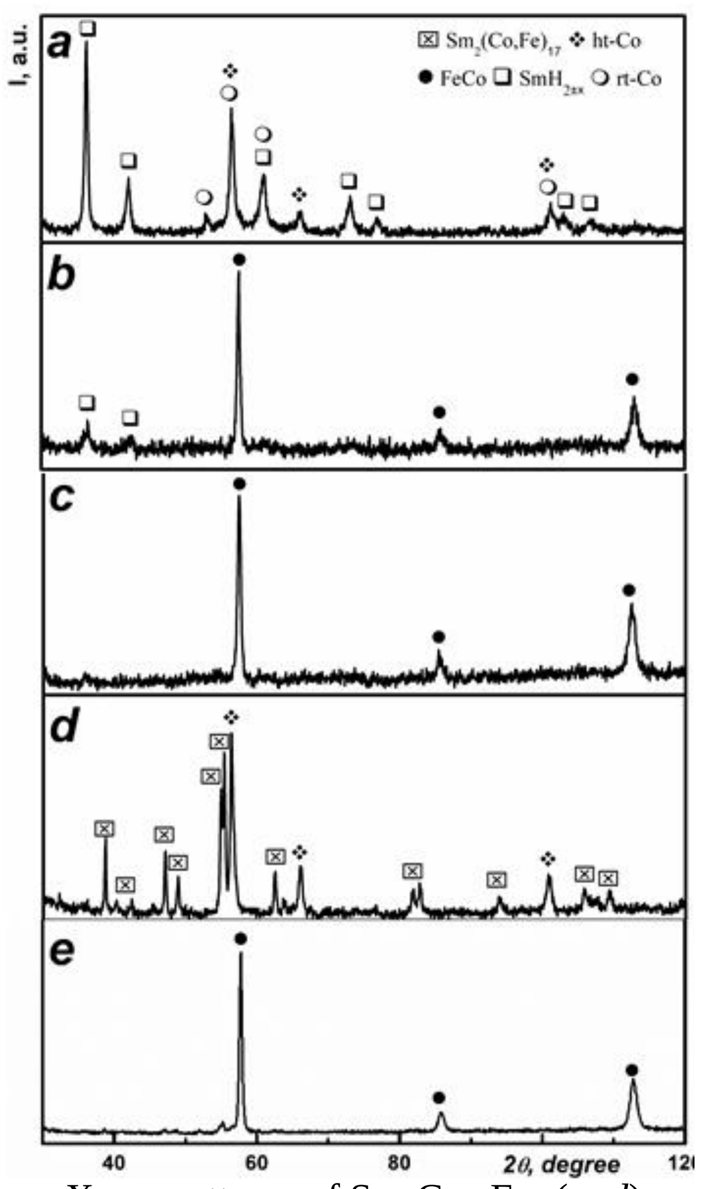
$Sm_2Co_{15}Fe_2$ (a, c) $Sm_2Co_9Fe_8$ (b, d)

Features of phase transformations during heating in hydrogen



The changes in hydrogen pressure (*1*) and DTA signal (*2*) during heating $Sm_2Co_{11}Fe_6$ (*a*) and $Sm_2Co_9Fe_8$ (*b*) alloys under PH_2 = 4 MPa up to 950 °C

X-ray phase analysis of the interaction products

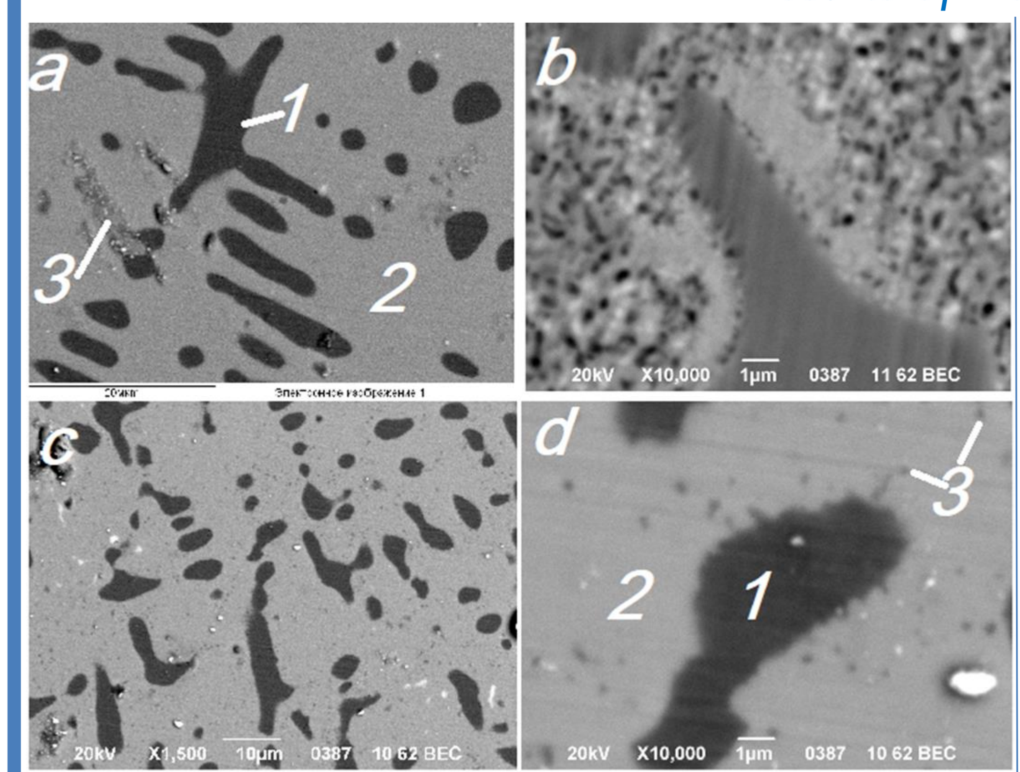


X-ray patterns of $Sm_2Co_{15}Fe_2$ (*a*, *d*), $Sm_2Co_{11}Fe_6$ (*b*), and $Sm_2Co_9Fe_8$ (*c*, *e*) alloys after heating under PH_2 = 4 MPa (*a*, *b*, *c*), and subsequent recombination in vacuum (*d*, *e*) at 950 °C

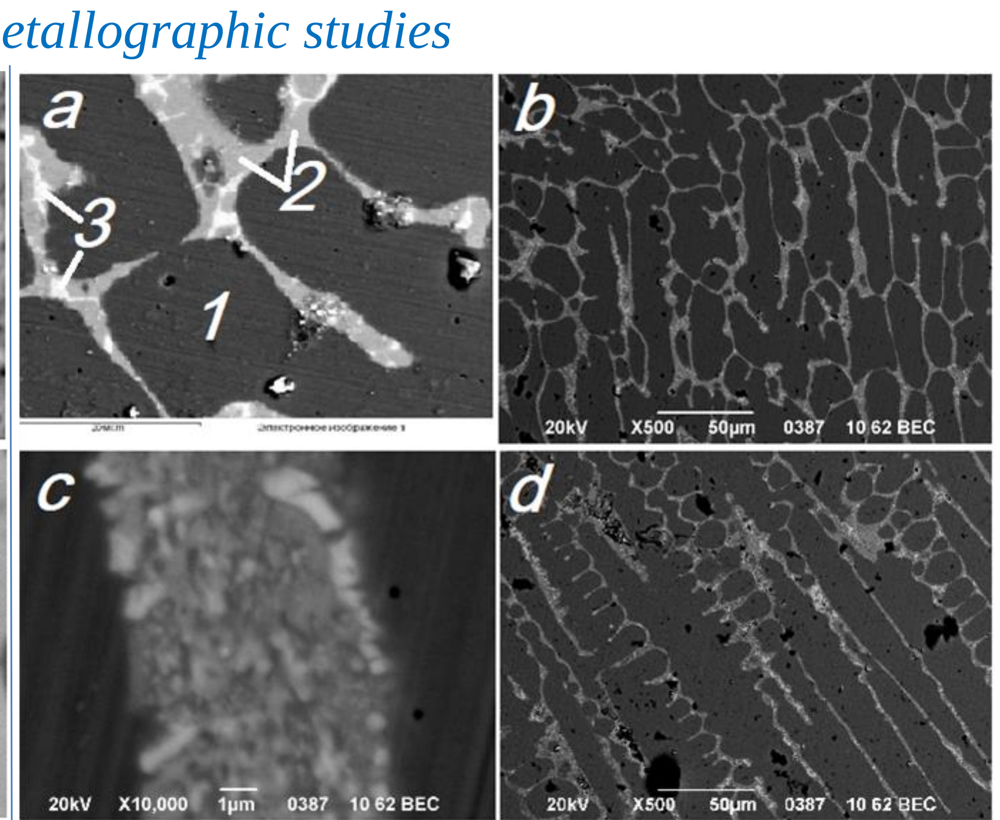
Table. Processing modes, phase composition and crystallographic parameters of $Sm_2Co_{17-x}Fe_x$ alloys (x = 2; 4; 6 and 8)

Alloy	Processing modes: Stage	PH_2, MPa	T_{max}, °C	Phase	The relative ratio of phases, vol. %	Lattice parameters: *a*, nm	*c*, nm
$Sm_2Co_{15}Fe_2$	Initial			Sm_2Co_{17} ht-Co	77 23	0.842(1) 0.3558(7)	1.226(3) –
	HD	2	950	ht-Co SmH_x rt-Co	25 14 61	0.353(1) 0.558(3) 0.251(1)	- - 0.407(4)
	HD	4	950	ht-Co $SmH_{2±x}$ β-Co	38 32 30	0.3543(1) 0.5396(1) 0.250(1)	– – 0.408(7)
	HD DR	4 vacuum	950 950	Sm_2Co_{17} ht-Co	46 54	0.841(1) 0.3559(3)	1.228(3) -
	HD DR	0.5 vacuum	950 950	Sm_2Co_{17} ht-Co	62 38	0.841(1) 0.3557(4)	1.226(3) -
$Sm_2Co_{13}Fe_4$	Initial			Sm_2Co_{17} FeCo	63 37	0.845(4) 0.2842(4)	1.233(7) –
	HD	4	950	ht-Co $SmH_{2±x}$ rt -Co	41 32 27	0.3544(4) 0.539(1) 0.250(10	– – 0.406(7)
$Sm_2Co_{11}Fe_6$	Initial			Sm_2Co_{17} FeCo	67 33	0.847(5) 0.2857(8)	1.243(8) –
	HD	4	950	$SmH_{2±x}$ FeCo	7 93	0.5365(3) 0.2847(1)	– –
	HD DR	4 vacuum	950 950	Sm_2Co_{17} FeCo	70 30	0.846(6) 0.2854(7)	1.236(9) -
$Sm_2Co_9Fe_8$	Initial			Sm_2Co_{17} FeCo	59 41	0.845(5) 0.2856(8)	1.250(8) –
	HD	4	950	FeCo	~100	0.2855(1)	–
	HD	0.5	950	FeCo	~100	0.2852(2)	–
	HD DR	0.5 vacuum	950 950	FeCo	~100	0.2840(2)	–

Results of metallographic studies



SEM microstructure of $Sm_2Co_{11}Fe_6$ alloy in the initial state (*a*) after disproportionation (*b*) and recombination (*c*, *d*)



SEM microstructure of $Sm_2Co_9Fe_8$ alloy in the initial state (*a*), after disproportionation (*b*, *c*) and recombination (*d*)

Conclusions

It was found that about 10 at. % Fe dissolves in the ferromagnetic phase $Sm_2(Co,Fe)_{17}$ of all investigated alloys. The composition of the soft magnetic phase depends on the content of the substituent element. Thus, the $Sm_2Co_{15}Fe_2$ alloy contains ht-Co, while all other alloys - the FeCo intermetallic. The iron content in the structural component based on cobalt increases from 14 at. % in the $Sm_2Co_{15}Fe_6$ alloy up to 50 at. % in the $Sm_2Co_9Fe_8$ alloy. The microstructures of $Sm_2Co_{17-x}Fe_x$ alloys with x = 2; 4 and 6 are similar: dendritic branching of magnetically soft phase (ht-Co or FeCo) in the matrix of the $Sm_2(Co,Fe)_{17}$ ferromagnetic phase. It has been showing that fully HDDR occurs in this alloys group, which leads to the crushing of the ferromagnetic phase and the formation of fine inclusions based on it. The FeCo intermetallic dominates in the microstructure of the $Sm_2Co_9Fe_8$ alloy, in which the releases of the ferromagnetic phase with the regions of the Sm-enriched phase are observed. The disproportionation of the ferromagnetic phase in this alloy is irreversible.

References

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