Central European Journal of Energetic Materials, **2008**, *5*(3-4), 37-44.
ISSN 1733-7178

Synthesis of Iron Aluminates and a New Modification of Alumina at Impact of Explosive

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Abstract: Synthesis of new high-strength superdispersed materials by use of detonation and shock waves is a developing branch of manufacture. Diamond, metal oxides, nitrides, and many other substances are produced and find industrial application.
The objective of current work was the method of synthesis of iron aluminates and a new modification of alumina. For the synthesis initiation of explosion of aluminized composite explosive by impact was firstly used.

Keywords: alumina oxides, iron aluminates, synthesis, explosive, impact structure

Introduction

Synthesis of new high-strength superdispersed materials by use of detonation and shock waves is an advanced technology. Diamond, metal oxides, nitrides, and many other substances are produced and find industrial application.

Lately, some modification of alumina, $Al_{8/3}O_4$, has been synthesized by detonation in explosive vacuum calorimeter [1-3]. Parameters of elementary cell of $Al_{8/3}O_4$, a = 7.935(1) and 7.953(3) Å, were similar to value, a = 7.948 for σ-Al_2O_3 melt crystallized [4]. The interplanar distances for both alumina phases were the same, but line strength of hkl=422, 511, 440 for new modification of alumina was differed from that for σ-Al_2O_3. It was concluded, that synthesized phase differed from σ-Al_2O_3.

By shock loading on $Al(OH)_3$ (Gibbsite) included in steel ampoule $FeAl_2O_4$ (Hercynite) has been synthesized [5]. Obviously iron aluminate formed at high pressure and temperature by reaction of decomposition products of aluminum hydroxide with steel wall of ampoule:

$$Al_2O_3 \cdot 3H_2O_{(cr)} \xrightarrow{P,t} \alpha\text{-}Al_2O_{3(cr)} + 3H_2O_{(g)}.$$

Most possible product of reaction of water steam with steel wall was wustite:

$$XFe + H_2O_{(g)} \xrightarrow{P,t} Fe_xO + H_{2(g)}.$$

X-ray diffraction study of reaction product showed, that very possibly Hercynite formed by reaction of corundum with wustite of stoichiometric composition:

$$FeO + \alpha\text{-}Al_2O_3 \xrightarrow{P,t} FeAl_2O_4$$

In this work the method of synthesis of iron aluminates and a new modification of alumina with structure of spinel derivative is described. For the synthesis initiation of explosion of aluminized composite explosive by impact was firstly used. The mechanism concept of explosion initiation by impact in explosives is given in work [6].

Experimental

Synthesis was carried out in steel tool (Figure 1) consisting of a muff, two cylindrical rollers by diameter, 10 mm and length, 11 mm. A sample was prepared by mixing high explosive with aluminum, mass of sample was 0.05 g, and the density was 0.9 g/cm^3. A sample was placed between rollers.

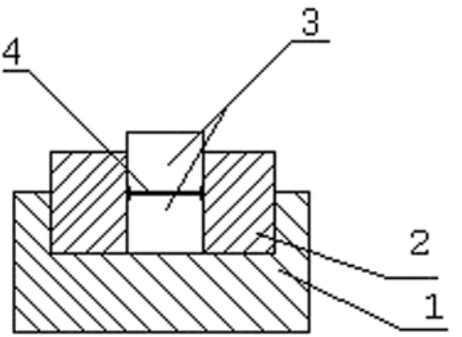

Figure 1. Experimental set: 1 – pallet; 2 –muff; 3 – rollers; 4 – sample.

Tools with sample of explosive mixture were placed on anvil of vertical fall hammer machine K-44-II. Falling weight was M = 10 kg, its height of falling down was H = 500 mm. The explosive transformation of substances accompanied with sound effect, flame or traces of burns on rollers or muff of the tools was accepted as the explosion.

After impact the sample was examined by X-ray diffraction (XRD) methods using $CuK\alpha_1$ radiation in chamber "Huber Imaging Plate Guiner Camera".

Results and Discussion

Composition of explosive is presented in Table 1. Calculated pressure of explosion in experiments was P = 1.0-1.5 GPa. Temperature of explosion was calculated by means of computer code REAL [7].

Table 1. Composition of explosive and calculated temperature of explosion

Sample	Composition of explosive, wt. %	Temperature, K	Number of tests
1	HMX/Al-90/10	3797	6
2	HMX/Al-70/30	3838	3
3	HMX/Al-80/20	4097	5
4	A-IX-2	4000	3
5	HMX/Al-60/40	3868	3

The analysis of XRD data given in Table 2 has shown that a three phases (two phases of iron aluminates and one phase of alumina) were formed as a result of explosion of the sample 1.

Table 2. The X-ray diffraction data of sample 1

I	Iron aluminates		Alumina	
	d_e, Å	hkl	d_e, Å	hkl
52	2.8537	220 220*	-	---
27	-	---	2.8000	220
75	2.4383	311	-	---
30	2.4156*	311	-	---
42	-	---	2.3851	311
17	-	---	2.2854	222
50**	2.0238	400	-	---
93	-	---	1.9795	400
13	1.6508	422	-	---
24	1.5564	511	-	---
13	1.5399*	511	-	---
20	-	---	1.5244	511
35**	1.4306	440	-	---
100	-	---	1.3996	440
9	-	---	1.1434	444

*$Fe_{0.5}Al_{2.33}O_4$ **Coincide with lines of aluminum and iron

The phase with parameter of an elementary cell, a = 8.090(4) Å of iron aluminates was presented at a considerable quantity. It is a solid solution of alumina with hercynite $Fe_{1-x}Al_{2+x}O_4$ on the base of data of work [8]. Hercynite also has close lattice parameter with a very high degree of the reversion of spinel $(Fe_{0.77}Al_{0.23})[Al_{1.77}Fe_{0.23}]O_4$ [9].

The hydrogen containing in a molecule of any explosive, practically completely turns to water at a detonation. The carbon depending on density of a charge can either completely be translated in CO_2, or be distributed between CO and CO_2.

The second variant can be in low density of charges. At detonation CO_2 and H_2O are active oxidizers of aluminum and iron. Carbon oxides does not react with aluminum, but it is capable (on the literary data) to restore iron aluminates at temperatures 800-1100 °C up to iron on reaction:

$$FeAl_2O_4+CO \rightarrow Fe+Al_2O_3+CO_2$$

The second phase of iron aluminates was present at small amount. Two most intensive lines of spinel with hkl 311 and 511 were fixed on X-ray photograph (in

Table 2 they are marked by an asterisk). These lines were used for account of an elementary cell parameter: $a = 8.002$ (2) Å. This size a little bit differs from given in work [10] for $Fe_{0.5}Al_{2.33}O_4$ and coincides with parameter a of $Zn_{0.54}Al_{2.31}O_4$ and $Zn_{0.33}Al_{2.45}O_4$ phases [11]. Obviously, phase of $Fe_{0.5}Al_{2.33}O_4$ is product of thermal disintegration $Fe_{1-x}Al_{2+x}O_4$ or $(Fe_{0.77}Al_{0.23})[Al_{1.77}Fe_{0.23}]O_4$ by reaction:

$$7FeAl_2O_4 = 6Fe_{0.5}Al_{2.33}O_4 + 4Fe + 2O_2$$

X-ray photograph of the third phase (alumina) is formally possible converting in spinel lattices with high parameter of an elementary cell $a = 7.920(1)$ Å.

Rentgenometric data of the sample 2 except two lines of small intensity (1.8225 and 1.1837 Å) are converting for spinel lattices with large parameter a – 7.926(2) Å too. X-ray photographs of the samples 3, 4 and 5 are converting in spinel lattices with $a = 7.933(3)$, 7.929(3) and 7.934(2) Å accordingly. The ratio of intensity lines of these phases is differ from σ-Al_2O_3 [12], and by parameter a - from γ-Al_2O_3. Completely the X-ray photograph of the sample 2 (Table 3) were possible converting in primitive hexagonal cell, derivate from face-centered cubic cell with $a = 9.151(1)$ Å, $c = 2\,c_o = 7.945(2)$ Å, (c_o – parameter of sub cell, 3.972 Å), $c/a = 0.868$, $V = 576$ Å^3. The groups $P\frac{6}{m}cc$, P6cc are possible. This hexagonal cell is interfaced with primitive tetragonal cell with twice smaller volume: $a = 7.941(2)$ Å, $c = 4.575(1)$ Å, $c/a = 0.576$, $V = 288$ Å^3. All lines of X-ray photograph of aluminas synthesized (Table 3) are converting in parameters of this cell, as well as in the case of hexagonal completely. It is possible to allocate a sub cell with $a_o = a/2 = 3.964$ Å and $c = 4.575$ Å in a tetragonal cell, as well as in hexagonal. All lines of X-ray photograph, except 1.8225 and 1.1837 Å are converting in these parameters.

Table 3. The X-ray diffraction data of samples

I	d_e, Å	hkl	
		variant I	variant II
28	2.8048	022	220
-	-	121	121
78	2.3916	122	221
16	2.2872	220	002
92	1.9816	040	022
-	-	222	040
8	1.8225	114	041
26	1.5250	330	003
100	1.4013	242	123
-	-	151	241
28	1.1837	036	360

We cannot directly convert from tetragonal lattices to hexagonal except variant, when the relation of parameters c/*a* in both lattices is equal or close to one. This variant takes place in this work. The matrix of vectors transformation of tetragonal cell to hexagonal looks like:

$$\begin{vmatrix} 0 & 0 & -2 \\ -1 & 0 & 1 \\ 0 & 1 & 0 \end{vmatrix}$$

The transition from hexagonal lattice to tetragonal is described by the following matrix:

$$\begin{vmatrix} -0.5 & 0.5 & 0.0 \\ 0.0 & 0.0 & 1.0 \\ -0.5 & -0.5 & 0.0 \end{vmatrix}$$

The similar case was observed in work [13]. The mechanism of transformation structure of high-temperature quartz type in structure of keatite in composition $LiAlSi_2O_6$ was observed in this work. This substance has two polymorphic modifications. One of them obtained by heating of glass of spodumen composition $Li_2O{\cdot}Al_2O_3{\cdot}4SiO_2$ and has a structure of high-temperature quartz type with a = 5.217 Å, c = 5.464 Å, c/a = 1.047, V = 128.8 Å^3. Second modification is formed by heating and has structure of keatite with tetragonal lattice: a = 7.541 Å and c = 9.156 Å, c/a = 1.214, V = 520.7 Å^3. The transition from tetragonal lattice to hexagonal with a = 5.298 Å, c = 5.332 Å, V = 129.6 Å^3 is described by the following matrix:

$$\begin{vmatrix} 0.25 & 0.25 & 0.50 \\ 0.25 & 0.25 & -0.50 \\ 0.50 & -0.50 & 0.00 \end{vmatrix}$$

In Table 3 interplanar distances of alumina synthesized is converting in two variants: parameters of hexagonal lattices (the first variant) and tetragonal lattices (the second variant). Converting was carried out in lattices of spinel (Tables 2 and 4). The results of converting are presented in Table 5 in two variants. If we assume, that the structure of synthesized alumina is close to $Al_{8/3}O_4$, then in the case of hexagonal lattice at Z = 9 calculated density was equal 3.527 g/cm^3. Absence of monocrystal data does not allow to make an unequivocal choice as between these lattices, and to define superstructure type.

Table 4. The X-ray diffraction data of samples

Sample 3		Sample 4		Sample 5		hkl
I	d_e, Å	I	d_e, Å	I	d_e, Å	
10	2.8022	10	2.8002	10	2.8024	220
8	2.3941	7	2.3928	9	2.3940	311
9	1.9830	5	1.9825	7	1.9844	400
2	1.5272	2	1.5255	2	1.5270	511
8	1.4021	6	1.4020	7	1.4023	440

Table 5. The X-ray diffraction data of samples

Sample	Parameters of an elementary cell, Å			
	hexagonal		tetragonal	
	a	c	*a*	c
1	9.145(3)	7.921(2)	7.916(3)	4.572(2)
2	9.151(1)	7.945(2)	7.941(3)	4.575(3)
3	9.161(5)	7.926(5)	7.931(1)	4.789(2)
4	9.155(5)	7.936(5)	7.929(4)	4.781(6)
5	9.161(5)	7.934(5)	7.933(3)	4.787(4)

Conclusion

Firstly, we synthesized cationic-defective iron aluminates $Fe_{0.5}Al_{2.33}O_4$ and new modification of alumina by impact of explosive containing aluminum. The structure of these new phases was determined by X-ray diffraction method.

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