Obrabotka metallov (tekhnologiya, oborudovanie, instrumenty) = Metal Working and Material Science. 2023 vol. 25 no. 3 pp. 126–136
ISSN: 1994-6309 (print) / 2541-819X (online)
DOI: 10.17212/1994-6309-2023-25.3-126-136

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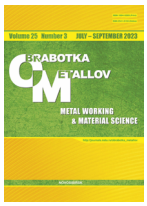

Synthesis of Ti–Fe intermetallic compounds from elemental powders mixtures

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ARTICLE INFO

Article history:
Received: 10 May 2023
Revised: 23 May 2023
Accepted: 21 June 2023
Available online: 15 September 2023

Keywords:
Powder mixture
Mechanical activation of synthesis
Thermal explosion
Titanium-Iron intermetallic compounds
Phase composition
Enthalpy of compounds formation

Funding
The work was supported by the Russian Science Foundation (project number 23-29-00106): "In situ synthesis of metal matrix composites with submicron carbide strengthening phase".

Acknowledgements
Research was partially conducted at core facility "Structure, mechanical and physical properties of materials".

ABSTRACT

Introduction. Intermetallic compounds Fe_2Ti and $FeTi$ are of practical application as hydrogen accumulators ($FeTi$) or as magnetic materials (Fe_2Ti). Due to the peculiarities of the double equilibrium diagram, the production of these intermetallic compounds by casting is difficult. Therefore, powder metallurgy methods are widely used combined with preliminary mechanical activation of the powder mixtures. **The aim of the work** is to investigate the possibility of obtaining single-phase compounds from powder mixtures of titanium and iron of target compositions. **Research methods.** Mechanically activated powder mixtures, products of combustion and subsequent annealing were studied by X-ray phase analysis, optical metallography, and scanning electron microscopy using elemental composition determination by energy-dispersive X-ray spectroscopy. **Research methodology.** Powder mixtures were mechanically activated for 20 minutes in an *Activator 2S* planetary ball mill with an intensity of 40 *g* and a ball/mixture ratio of 20. The mechanically activated mixtures were heated in a sealed reactor in argon media at an average rate of 85 C°/min. **Results and discussion.** At a temperature of about 500 °C, thermographs of thermocouples placed in a mechanically activated mixture showed a sharp rise (thermal explosion), indicating an exothermic reaction in the mixture. The rise for the $2Fe + Ti$ composition turned out to be more pronounced than that for the $Fe + Ti$ composition. X-ray diffraction analysis showed that the main reaction product is the Fe_2Ti compound for both mixtures. The predominant formation of Fe_2Ti is explained by the greater negative enthalpy of Fe_2Ti formation of compared to $FeTi$ (−87.45 and −40.58 kcal/mol, respectively). **Conclusion.** High-temperature annealing of thermal explosion products did not make it possible to obtain single-phase target products. The content of secondary phases and unreacted reagents changed little after annealing. Based on the obtained results, it was concluded that the thermodynamic factor (the enthalpy of formation of the intermetallic compound) is the main one that determines the phase composition of the synthesis products in powder mixtures of titanium and iron.

For citation: Pribytkov G.A., Baranovskiy A.V., Korzhova V.V., Firsina I.A., Krivopalov V.P. Synthesis of Ti–Fe intermetallic compounds from elemental powders mixtures. *Obrabotka metallov (tekhnologiya, oborudovanie, instrumenty) = Metal Working and Material Science*, 2023, vol. 25, no. 3, pp. 126–136. DOI: 10.17212/1994-6309-2023-25.3-126-136. (In Russian).

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Introduction

There are two intermetallic compounds on the iron-titanium equilibrium diagram: Fe_2Ti and *FeTi* (fig. 1). Iron monotitanide *FeTi* is the most studied among the intermetallic compounds of the iron-titanium system. This compound is known as a perspective hydrogen storage material. *FeTi* is inferior to the most promising magnesium alloys and alloys of rare-earth metals in terms of hydrogen capacity, ability to pass into an active state with respect to hydrogen sorption, kinetic characteristics of sorption-desorption and cyclic stability [1]. However, due to the low cost of raw materials, attempts continue to obtain a material with improved sorption characteristics. In this case, iron and titanium powders are used as raw materials, and mechanical activation (*MA*) of synthesis is used as a method for obtaining the material during long-term processing of the powder mixture in planetary mills.

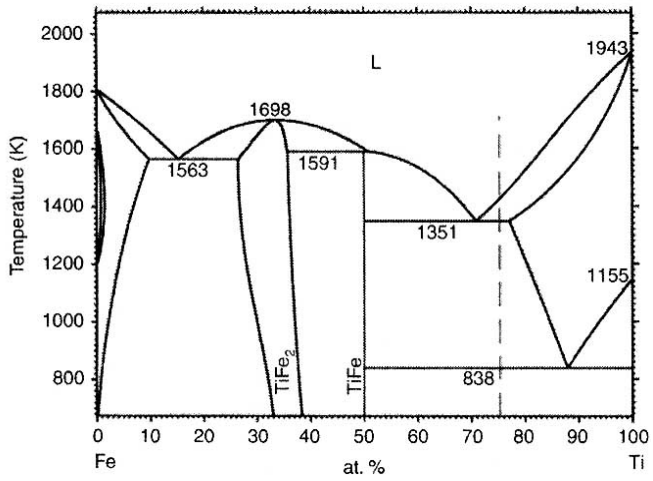


Fig. 1. Phase diagram of the Fe-Ti system

Intensive studies of the behavior of iron-titanium powder mixtures during mechanical activation began in the early 2000s. It was found out in [2] that during long-term (up to 92 hours) processing of mixtures in a *SpexMixer/MillModel 8000* magnetic vibratory mill, full amorphization of titanium and iron is completed. The formation of intermetallic compounds was not observed in this case. Extensive studies of the sorption properties of materials obtained by mechanical activation of iron and titanium powder mixtures were studied by *A.V. Zadorozhny* and colleagues [3–8]. For mechanical activation, an *AGO-2S* planetary mill was used. The processing was carried out in an argon media at a rotation speed of 840 rpm. The phase composition of the mechanical synthesis' products depended on the dispersity of titanium and iron powders. The formation of intermetallic compounds was not observed when using coarse powders, i.e. 280 and 450 μm, and processing time of 120 minutes [3]. When 5–10 μm fine titanium powder was used, the product obtained by processing of an equiatomic mixture for 30 minutes at the same intensity, i.e. 840 rpm, consisted of single-phase *FeTi* [8]. This result is in conflict with the results of [2], in which no intermetallic compound was formed even after 92 hours of processing in a *SpexMixer/MillModel 8000* mill. The authors of [8] explain the reason for this mismatch by the low intensity of *MA* in the mill used in [2]. Pressed *FeTi* single-phase powder samples with a nanocrystalline structure during thermal cycling in a hydrogen medium retained its shape without cracking due to the formation of bridges that hold adjacent particles together [4, 7]. In order to improve the sorption properties of *FeTi* obtained by intensive *MA*, various powder additives were added into the mixture of titanium and iron: 20 at. % *Al* or 6 at. % *Cr* [6], 1 at. % *S* or 2 at. % *Mg* [5]. It was shown, that these additives improve the sorption characteristics – simplification of the procedure for activating hydrogen uptake and a decrease in the pressure of the plateau area.

In a the brief follow-up review [9], hydrogen storage alloys based on magnesium and rare earth metals such as *FeTi* have improved sorption characteristics in the nanocrystalline state. Along with mechanical activation, to obtain nanocrystalline intermetallic compounds of the iron-titanium system, attempts are made to use other methods, in particular, severe plastic deformation of powder mixtures in a *Bridgman anvil* [10]. To create a nanocrystalline structure, it looks perspective to intensively grind *FeTi* powder, previously obtained by casting or using powder technologies. The most technologically simple way to obtain intermetallic compounds is the synthesis in iron-titanium powder mixtures of binary intermetallic compounds [11, 12]. This synthesis can be realized either directly in the process of mechanical activation [12] or during the subsequent initiation of the reaction in mechanically activated mixtures [13, 14]. In [15], *FeTi* and Fe_2Ti compounds were obtained from powder mixtures of the respective compositions. Since the mixtures were not subjected to preliminary mechanical activation, it becomes possible to initiate

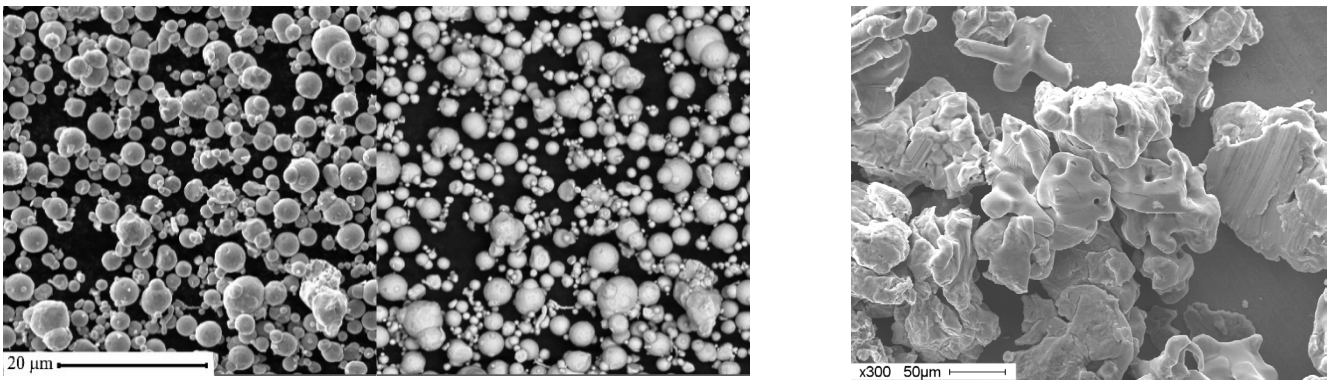
synthesis in the wave mode only with preliminary heating of the compacts to temperatures over 700 °C. In [16], mixtures corresponding to the compositions of binary intermetallic compounds were subjected to short-term, i.e. up to 10 minute duration, intense *MA* at subsequent rapid heating, i.e. about 300 K/min. The synthesis reaction in the thermal explosion mode was initiated in the 450–500 °C range depending on *MA* time and the mixture composition. In [17], the *Fe*-20 % *Ti* mixture was processed for 4 and 20 minutes in a planetary mill at intensity close to that used in [16]. The formation of the intermetallic compounds was not detected even when the *MA* mixture was annealed at 500 °C. A possible reason for the mismatch in the results of [16] and [17] is the different heating rates of the *MA* mixtures. With rapid heating, the reaction surface is not contaminated by reaction diffusion products at the stage of slow heating to the annealing temperature, and the synthesis reaction occurs in the thermal explosion mode.

Thus, the problem of developing technological modes of mechanical activation of powder mixtures and the subsequent synthesis of single-phase intermetallic compounds of the titanium-iron system remains unresolved. The tasks of this study are to investigate the phase composition of the synthesis' products in mechanically activated powder mixtures of titanium and iron, and to investigate the possibility of obtaining single-phase binary intermetallic compounds. Two compositions were used, corresponding to *FeTi* and Fe_2Ti compounds. The Fe_2Ti intermetallic is also of practical interest as a magnetic material [18].

Research methodology

The reaction mixtures were prepared from titanium powder with a dispersity of less than 160 μm and iron powder with a dispersity of less than 5 μm. The morphology of the powders is shown in fig. 2. The powders' weighted portions of 15 grams were mixed for 4 hours in a mixing tank and placed in the jars of an *Activator-2S* planetary ball mill. To prevent sticking of the powders to the balls and walls, 0.5 cm^3 of alcohol was injected into each jar. Mechanical activation was carried out at a jar rotation speed of 755 rpm (centrifugal acceleration 40 g). A weight ratio of steel grinding balls with a diameter of 6 mm to the reaction mixture was equal to 20. In total, every mixture was mechanically activated for 20 minutes. To prevent the Activator overheating, the jars' rotation was stopped every 10 minutes, for 10 minutes to cool the jars with water flow.

The mechanically activated mixtures were poured into cylindrical titanium containers and slightly compacted. The containers were placed into a sealed reactor. A design of the reactor is presented in [19]. The reactor was lowered in the furnace preheated up to 800 °C. The reactor was continuously purged with argon at a flow rate of 4 L/min. The temperature was registered automatically by two thermocouples. The junction of the first thermocouple, thermally insulated from the furnace radiation with a layer of asbestos, was located on the outer wall of the reactor. The junction of the second thermocouple was located in the



a *b*

Fig. 2. SEM images of the initial powders morphology:

a – *VM* iron* (left side of the photo – back scattered electron image (BSE), right one – secondary electron image (SE)); *b* – TPP-8 titanium**

* TC 6-09-2227-81 «Reduced metallic iron»; ** TC 1791-449-05785388-99 «Titanium sponge powder»

container with the reaction mixture. The ignition temperature T_{ign} and the maximum combustion temperature T_{max} were processed from the recorded reaction mixture temperature curve. The reactor was removed from the furnace after equalization of temperatures of the outer reactor surface and the powders mixture. Equalization happened in about 2–4 minutes after the temperature peak appearance. Then the reactor was cooled in air. Part of the reacted powder mixtures were annealed in vacuum of 0.01 Pa with variation of the temperature and annealing duration.

Mechanically activated powder mixtures, products of combustion and subsequent annealing were studied using the equipment of the S*hared Use Center "Nanotech"* of the ISPMS SB RAS by X-ray phase analysis (*DRON-7* diffractometer, Burevestnik, Russia). The phases were identified based on the results of X-ray diffraction analysis using the *ASTM* X-ray database, and the primary results were processed using the *RENEX* and *PDWIN* software programs.

Results and discussion

Fig. 3 shows thermographs of *MA* reaction mixtures of two compositions at an average rate of 85 ± 5 deg/min and the time derivatives of temperature (the rate of increase in mixture temperature). There is an abrupt increase in temperature at 500–530 °C as a result of self-ignition, that is, the reaction occurs in the thermal explosion (*TE*) mode (fig. 3a, b). The duration of heating to self-ignition was 6–7 minutes. The rate of temperature elevation after ignition and the peak value for the *2Fe + Ti* mixture turned out to be much higher (fig. 3a, c) than for the equiatomic mixture (fig. 3b, d). The reason is twice the negative value of the enthalpy of formation of the Fe_2Ti compound compared to that for *FeTi*: –87.45 and –40.58 kcal/mol, respectively [20].

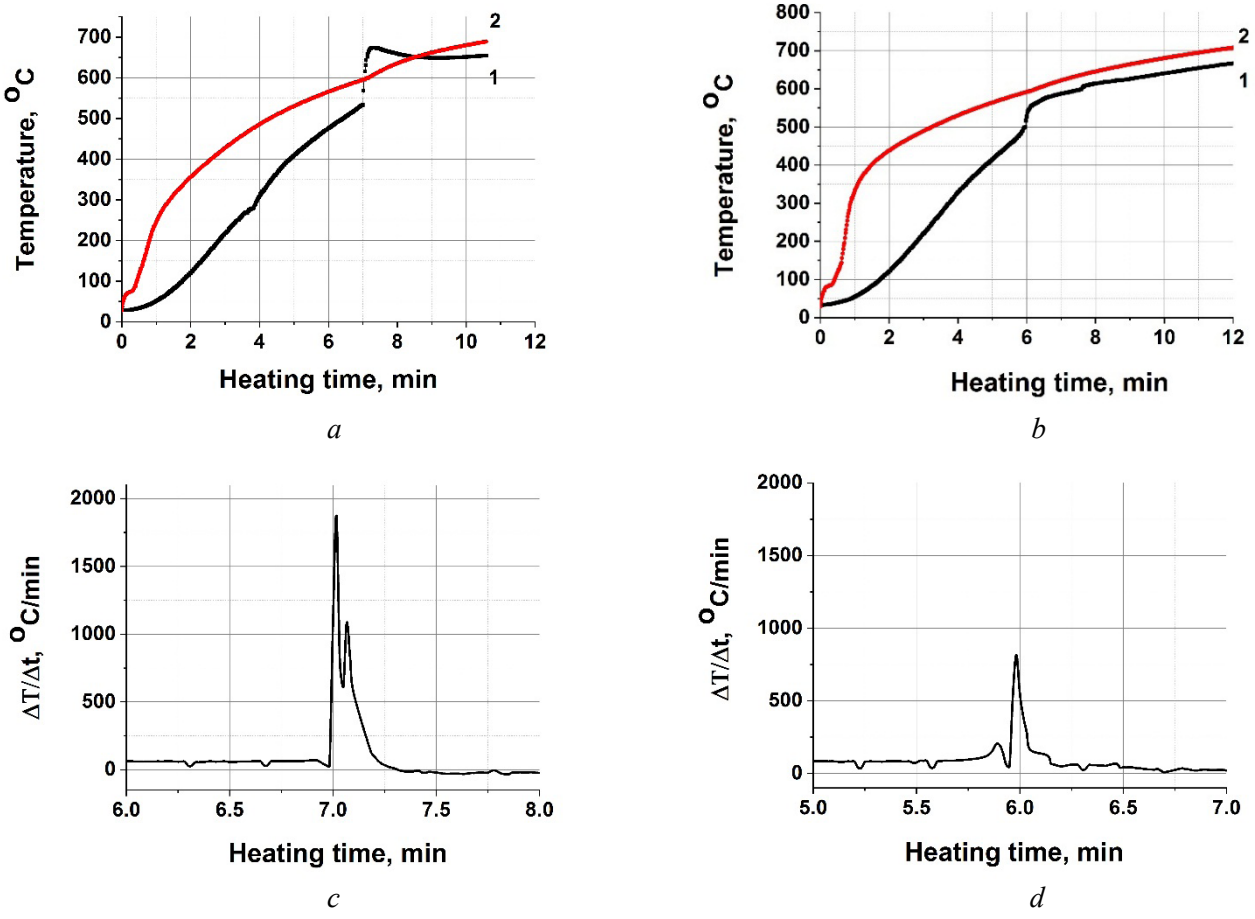


Fig. 3. Heating thermographs (*a*, *b*) and rate of temperature change (*c*, *d*) of mechanically activated powder mixtures: *2Fe + Ti* (*a*, *c*) and *Fe + Ti* (*b*, *d*):

1 – the sample temperature; *2* – temperature of the outer surface of the reactor

According to the results of X-ray phase analysis (fig. 4, Table 1), after 20 minutes of mixtures processing at the selected intensity, the phase composition of the mixtures remains indentical. That means mechanical synthesis of intermetallic compounds does not take place. In the products of a thermal explosion, a small content of intermetallic compounds is determined (at the sensitivity level of the method). In this case, the Fe_2Ti compound is also present in the combustion products of the *Fe + Ti* composition in an amount approximately equal to the content of the target *FeTi* compound. The result correletes with the thermal explosion thermographs (fig. 3). Apparently, the formation of Fe_2Ti, which has a large negative enthalpy, is the reason for the low observable thermal explosion in a *Fe + Ti* mixture.

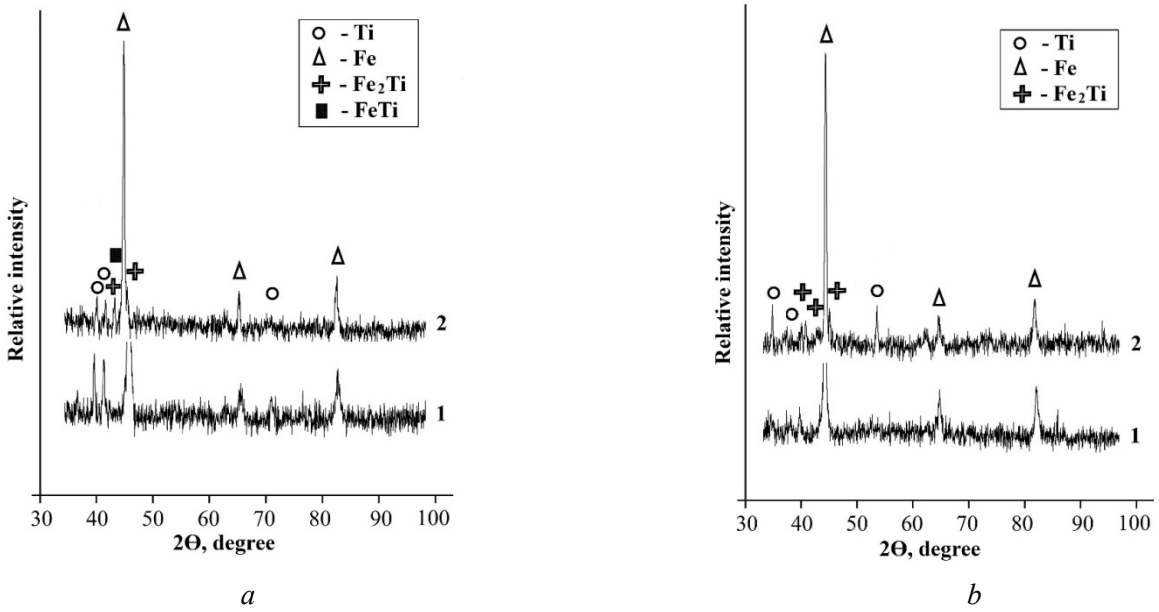


Fig. 4. X-ray patterns of mechanically activated mixtures (*1*) and thermal explosion products (*2*) in *Fe + Ti* (*a*) and *2Fe + Ti* (*b*) mixtures

Table 1

Phase composition of the mechanically activated (*MA*) mixtures and the thermal explosion (*TE*) products

Mixture composition	Mixture processing	Phase volume content, %			
		Fe (6-696)	*Ti* (5-682)	*FeTi* (19-636)	Fe_2Ti (15-336)
Fe + Ti	*MA*	70.7	29.3	–	–
Fe + Ti	*MA + TE*	71.4	12.9	6.6	9.1
2Fe + Ti	*MA*	75.1	18.7	–	6.2
2Fe + Ti	*MA + TE*	72.0	23.1	–	4.9

Since synthesis under thermal explosion conditions in *MA* mixtures gives an insignificant volume of target products, powder mixtures of both compositions (including those without preliminary *MA*), as well as thermal explosion products, were annealed in vacuum in order to establish technological conditions (temperature and time), providing the maximum yeild of target products. The results of detected phase composition after annealing are shown in Table 2. Due to the superposition of lines of different phases, the quantitative detection of the phase content is complicated. Therefore, the table contains tne approximate indications which obviously show that the Fe_2Ti intermetallic compound is the main phase in all cases, regardless of the mixtures composition the and the heat treatment mode. However, we failed to achieve a single-phase state of Fe_2Ti in annealed mechanically activated *2Fe + Ti* mixtures. After annealing, a

Table 2

Phase composition of mechanically activated mixtures after additional annealing in vacuum depending on the degree of compaction

Mixture composition	Modes of compaction and heat treatment of MA mixture		Volume content of phases, %					
	Compaction	Heat treatment	Fe_2Ti (15-336)	$FeTi$ (19-636)	Fe (6-696)	Ti (5-682)	$TiN_{0.9}$ (31-1403)	?
2Fe + Ti	Loose mixture	1,000 °C, 1 hour	73.3	2.2	24.5	–	–	–
		1,150 °C, 2 hours	74.5	4.8	20.7	–	–	–
		1,250 °C, 2 hours	73.2	3.1	23.7	–	–	–
	Compacted sample	1,250 °C, 2 hours	84	–	16	–	–	
Fe + Ti	Loose mixture	1,000 °C, 1 hours	89.5	7.9	–	0.7	–	1.9
		1,150 °C, 2 hours	83.4	4.5	–	–	7.4	4.7
		1,250 °C, 2 hours	84.0	1.1	–	6.8	4.5	1.6
	Compacted sample	1,250 °C, 2 hours	85	15	–	–	–	–

significant amount of unreacted iron remains. It can be explained by a sufficiently wide region of homogeneity of the Fe_2Ti compound (fig. 1). In annealed *Fe + Ti* mixtures, the Fe_2Ti phase is also the main phase, while the content of the target *FeTi* phase does not exceed 7.9 vol. %.

Perhaps, one of the reasons for the multiphase nature of the annealing products is the mechanically activated mixtures were annealed in a loose mixture. In order to increase the specific reactive surface area, the mechanically activated mixtures were pressed, and the compacts were subjected to high-temperature diffusion annealing under the same conditions as the mixtures in loose mixture.

The phase composition of the annealed compacts (Table 2) differs from the phase composition of the mixtures in loose mixture by the absence of undetermined phases, unreacted titanium and titanium nitride, the small content of which is determined in the annealed mixtures. However, there were no qualitative changes in the phase composition. The Fe_2Ti intermetallic compound remained the main phase in the compacts of both compositions. At the same time, the content of the target phases increased due to a decrease in the content of unreacted iron (for the *2Fe + Ti* composition) and secondary-side phases (for the *Fe + Ti* composition).

Conclusions

1. In mechanically activated powder mixtures of compositions corresponding to Fe_2Ti and *FeTi* binary intermetallic compounds, exothermic reactions occur under heating, causing temperature elevation and the formation of a small amount of intermetallic compounds.

2. Under the modes of mechanical activation of mixtures used in the study it is impossible to synthesize single-phase intermetallic compounds in thermal explosion reaction.

3. The predominant formation of the Fe_2Ti compound is explained by a thermodynamic stimulus: the negative enthalpy of Fe_2Ti formation is twice that of the *FeTi* compound.

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Conflicts of Interest

The authors declare no conflict of interest.