



UDC 669.018.25.017

<https://doi.org/10.17073/1997-308X-2024-4-17-25>

Research article

Научная статья



Experimental study of the feasibility of producing materials based on the metastable phase Ti_2Fe through explosive compaction and heat treatment

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Abstract. The primary regularities in the formation of the structure and phase composition of Fe–Ti system materials, which are promising for hydrogen storage under explosive compaction of titanium and iron powder mixtures, are considered. It has been established that under a loading regime ensuring shock-wave compression pressure $P = 11.5$ GPa and heating in the falling shock wave to 777°C , the powder mixture is compacted to an almost non-porous state due to the uniform plastic flow of particles in a direction perpendicular to the direction of shock compression. Under more severe loading conditions ($P = 12.5$ GPa and $t = 831^\circ\text{C}$), a monolithic state is also achieved, but the deformation character of the powder mixture component particles changes fundamentally: plastic deformation of the particles is localised in their surface layers and has a pronounced jet character with the formation of specific “vortices”. The influence of the plastic deformation mechanism of powder particles on the formation process of the metastable intermetallic phase Ti_2Fe with increased hydrogen capacity has been discovered. It has been established that solid layers of Ti_2Fe up to $20\ \mu\text{m}$ thick are formed at the contact boundaries of iron and titanium particles only in the case of jet flows of surface layers of particles. It has been shown that the cause of this effect is the local heating of the contact areas to a temperature above 1085°C , which according to the phase diagram of the Ti–Fe system, is the minimum temperature for the existence of a liquid phase in it. It has been demonstrated that an effective method for producing materials based on Ti_2Fe is the combination of explosive compaction of Fe and Ti powder mixtures and subsequent heat treatment with heating to 1100°C (reactive sintering in the presence of a liquid phase).

Keywords: explosive powder compaction, metastable intermetallic Ti_2Fe , hydrogen capacity, heat treatment, reactive sintering in the presence of a liquid phase

Acknowledgements: This research was supported by Volgograd State Technical University under the scientific project No. 6/464-22.

For citation: Krokhalev A.V., Kharlamov V.O., Chernikov D.R., Kuzmin S.V., Lysak V.I. Experimental study of the feasibility of producing materials based on the metastable phase Ti_2Fe through explosive compaction and heat treatment. *Powder Metallurgy and Functional Coatings*. 2024;18(4):17–25. <https://doi.org/10.17073/1997-308X-2024-4-17-25>

Экспериментальное исследование возможности получения материалов на основе метастабильной фазы Ti_2Fe с помощью взрывного прессования и термической обработки

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Аннотация. Рассмотрены основные закономерности формирования структуры и фазового состава материалов системы Fe–Ti, перспективных для хранения водорода, при уплотнении взрывом порошковых смесей титана и железа. Установлено, что при использовании режима нагружения, обеспечивающего давление ударно-волнового сжатия $P = 11,5$ ГПа и разогрев в падающей ударной волне до 777 °C, происходит уплотнение порошковой смеси до практически беспористого состояния за счет равномерного пластического растекания частиц в направлении, перпендикулярном направлению ударного сжатия. При более жестком нагружении ($P = 12,5$ ГПа и $t = 831$ °C) также достигается монолитное состояние, но характер деформации частиц компонентов порошковой смеси принципиально изменяется: пластическая деформация частиц локализуется в их поверхностных слоях и имеет ярко выраженный струйный характер с образованием специфических «завихрений». Обнаружено влияние механизма пластического деформирования частиц порошка на процесс образования метастабильной интерметаллидной фазы Ti_2Fe с повышенной водородной емкостью. Установлено, что сплошные прослойки Ti_2Fe толщиной до 20 мкм формируются на границах контактирования частиц железа и титана лишь в случае реализации струйных течений поверхностных слоев частиц. Показано, что причиной подобного эффекта является локальный разогрев приконтактных областей до температуры выше 1085 °C, являющейся, в соответствии с диаграммой состояния системы Ti–Fe, минимальной для существования в ней жидкой фазы. Показано, что эффективным методом получения материалов на основе Ti_2Fe является совмещение взрывного прессования смеси порошков Fe и Ti и последующей термической обработки с нагревом до 1100 °C (реакционное спекание в присутствии жидкой фазы).

Ключевые слова: прессование порошков взрывом, метастабильный интерметаллид Ti_2Fe , водородная емкость, термическая обработка, реакционное спекание в присутствии жидкой фазы

Благодарности: Исследование выполнено при финансовой поддержке ВолГТУ в рамках научного проекта № 6/464-22.

Для цитирования: Крохалев А.В., Харламов В.О., Черников Д.Р., Кузьмин С.В., Лысак В.И. Экспериментальное исследование возможности получения материалов на основе метастабильной фазы Ti_2Fe с помощью взрывного прессования и термической обработки. *Известия вузов. Порошковая металлургия и функциональные покрытия*. 2024;18(4):17–25.
<https://doi.org/10.17073/1997-308X-2024-4-17-25>

Introduction

The development of hydrogen energy imposes ever-increasing demands on hydrogen storage systems [1]. One of the most promising and safe methods for hydrogen storage is the use of hydride-forming intermetallics [2]. For example, the volumetric density of hydrogen in hydrides based on intermetallics ranges from approximately 60 to 100 kg/m³, whereas in the gaseous state, even at a pressure of 400 bar, it is only about 20 kg/m³ [3]. Such high values of hydrogen capacity enable the creation of anode materials for metal hydride batteries with a discharge capacity reaching 400 mA·h/g [1].

In addition to the currently most widespread intermetallic $LaNi_5$, one of the most attractive materials for

hydrogen storage is the intermetallic $TiFe$, which is characterized by low cost and the ability to operate at low pressures and ambient temperatures [1].

Currently, many researchers [4–11] are focused on studying the possibility of solving the “activation” problem of this material [12–15]. The issue is that $TiFe$ is very sensitive to air and forms a passivating layer that prevents hydrogen absorption. The classical activation method involves heat treatment, which consists of cyclic exposure to elevated and room temperatures at high hydrogen pressure [16]. However, other methods are now considered more promising, such as mechanical treatment (ball milling, cold rolling, high-pressure torsion) [8; 17–20], the use of increased (compared to stoichiometric) Ti content [5; 9], and

the addition of alloying elements such as Mn, Cr, Zr, Y, etc. [6; 10; 21–23].

When using an excess of Ti and alloying, it was found [5; 6; 9; 10; 21–23] that in addition to TiFe, a solid solution based on β -Ti (referred to in some works as the BCC phase or Ti_4Fe phase) and the intermetallic Ti_2Fe appear in the structure of the material, which act as a kind of “gate” for hydrogen [6–8]. Several studies [4; 5; 9–11] also note a general increase in the hydrogen capacity of materials during primary hydriding, indicating hydrogen absorption not only by the main TiFe phase but also by the secondary β -Ti and Ti_2Fe phases. Statistical analysis of data obtained by various authors for three-phase materials based on TiFe, which do not require activation and contain excess amounts of Ti and/or Mn and Zr, showed that their hydrogen capacity during primary hydriding follows the law of additivity and can be described by the equation [24; 25]

$$H = 0.01537[\text{TiFe}] + 0.03213[\text{Ti}_2\text{Fe}] + 0.03847[\text{Ti}_4\text{Fe}],$$

where H is the hydrogen capacity of the material, wt. %; $[\text{TiFe}]$, $[\text{Ti}_2\text{Fe}]$, and $[\text{Ti}_4\text{Fe}]$ – are the contents of the respective phases, wt. %.

As follows from the given equation, the hydrogen capacity of the TiFe phase in the multiphase TiFe– Ti_2Fe – Ti_4Fe material is about 1.54 wt. % H (which is quite close to the experimental value of 1.70–1.85 wt. % H for the TiFeH hydride given in [10]), the capacity of the BCC solid solution Ti_4Fe is 3.85 wt. % H (slightly exceeding the known capacity of 3.7 wt. % H for the Ti_4FeH_8 hydride [9]), and the capacity of Ti_2Fe is about 3.21 wt. % H (also slightly higher than the theoretical estimate of 3.09 wt. % H for the $\text{Ti}_2\text{FeH}_{4.75}$ hydride given in [9]). It is interesting to note that in the presence of secondary β -Ti and Ti_2Fe phases, the main TiFe phase apparently does not form the TiFeH_2 hydride with the maximum possible capacity of 1.8–1.98 wt. % H [10], which is more than compensated by the contribution of β -Ti and Ti_2Fe .

Another important fact from the statistical analysis [24; 25] and consistent with the conclusions of [9] is that increasing the content of β -Ti in Ti and Fe-based materials reduces their reversible hydrogen capacity, indicating that the saturation of this phase with hydrogen is irreversible. The ability of the intermetallic Ti_2Fe to release stored hydrogen depends on the content of alloying elements: in their absence or with a small amount of Mn, hydrogen saturation is reversible [25], but with Zr alloying, ensuring maximum Ti_2Fe content in the material's structure, it is irreversible [9; 25]. This

leads to the conclusion that the most promising way to improve Ti–Fe system materials, capable of doubling their reversible hydrogen capacity, is to increase the Ti_2Fe content in their structure without using alloying. However, known attempts to solve this problem, based on melting the components followed by annealing [16; 26; 27], have failed [5].

This work explores the possibility of using explosive compaction of iron and titanium powder mixtures for this purpose. The starting point for choosing this method was the well-known fact of frequent intermetallic formation during explosion welding of steels and titanium alloys [28; 29], as well as the fact that during explosive compaction of powders and explosion welding, the area of intense plastic deformation on the surface of the joined components forms similarly [30; 31]. Using powder significantly increases the surface area in the material volume and reduces the effective diffusion paths of elements during interfacial interactions.

Materials and methods

To produce the materials, titanium powder grade PTM-1 and PZhV powder with particle sizes up to 200 μm from a commercial supplier in as-received condition were used. The Ti powder particles had a spongy shape, while the Fe particles were rounded with a pronounced polycrystalline structure (Fig. 1, *a*). The component content in the powder mixture was 36 wt. % Fe and 64 wt. % Ti, which is nearly stoichiometric for Ti_2Fe (see Fig. 1, *b*) and ensures equal volumetric content of Fe and Ti in the mixture.

Explosive compaction was performed by placing the initial powder mixture on the surface of a steel substrate and loading it with a flat, normally incident detonation wave through an intermediate layer separating the detonation products from the powder (Fig. 2). The calculation of the physical compression parameters implemented in the experiments was performed using the (P, U) -diagram method [33]. The calculation results are presented in the Table.

The phase composition, structure, and chemical composition of the phases of the obtained samples were investigated by X-ray phase analysis using a D8 Advance X-ray diffractometer (Bruker Optik GmbH, Germany) and scanning electron microscopy using a Versa 3D microscope (FEI, Czech Republic) with an integrated Apollo-X energy-dispersive X-ray microanalysis system (EDAX, USA).

Experimental results

The study of the materials obtained through explosive compaction showed that using this method,

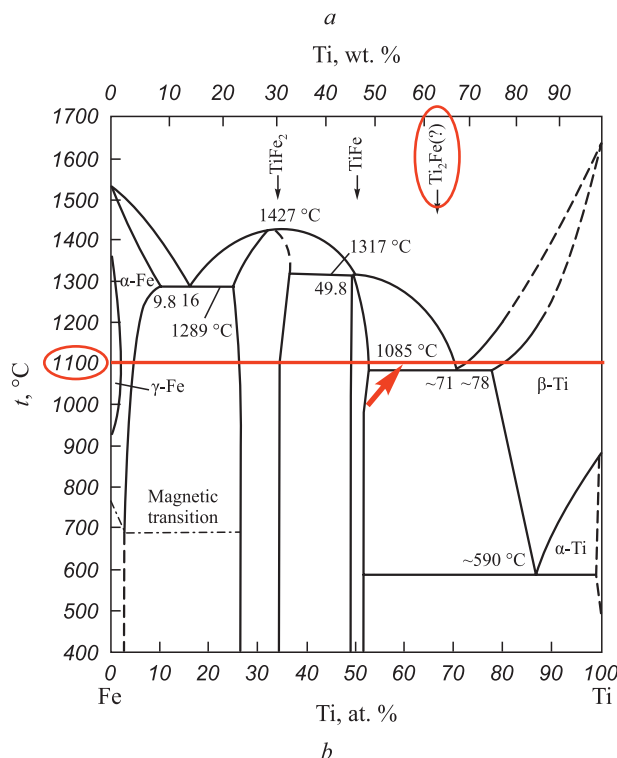
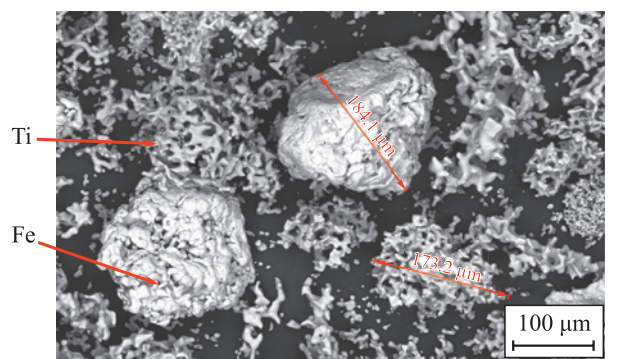


Fig. 1. View of Fe and Ti particles in the initial powder mixture (a) and phase equilibrium diagram (b) in the Fe–Ti system [32]

Рис. 1. Вид частиц Fe и Ti в исходной порошковой смеси (a) и диаграмма (b) стабильного фазового равновесия в системе Fe–Ti [32]

which ensures a shock-wave compression pressure of $P = 11.5$ GPa and a heating temperature up to 777 °C, the powder mixture is compacted into an almost monolithic material (Fig. 3, a). In this process, the deformation of the particles occurs due to uni-

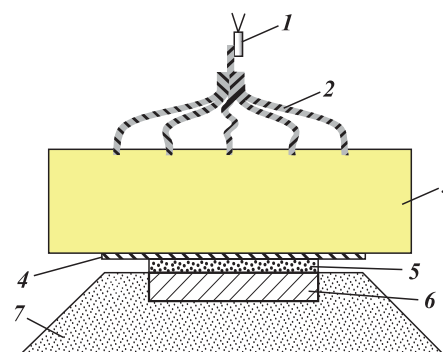


Fig. 2. Scheme of explosive loading [33]

1 – electric detonator; 2 – detonating cord; 3 – explosive charge; 4 – intermediate gasket; 5 – powder; 6 – substrate; 7 – sand pad

Рис. 2. Схема взрывного нагружения [33]

1 – электродетонатор; 2 – детонирующий шнур; 3 – заряд взрывчатого вещества; 4 – промежуточная прокладка; 5 – порошок; 6 – подложка; 7 – песчаная подушка

form plastic flow, resulting in noticeable flattening in the direction of shock compression and spreading in the transverse direction. No traces of changes in the initial phase composition were detected (Fig. 3, c). Even in the immediate vicinity of the interfacial boundaries, the chemical composition of the phases remained practically unchanged (Fig. 3, b).

Under more severe shock-wave compression conditions ($P = 12.5$ GPa and $t = 831$ °C) the deformation character of the component particles of the powder mixture changes fundamentally. Plastic deformation localizes in their surface layers and exhibits a pronounced jet character with the formation of specific “vortices,” as described in detail in [33; 35].

At the interfacial surfaces, solid layers up to 20 μm thick of an intermetallic compound (Fig. 4, b) are formed, which, based on its chemical composition (Fig. 4, c) and crystalline structure (Fig. 4, d), can be identified as the metastable phase Ti_2Fe .

Detailed examination of the microstructure in the interaction zone of the original powder mixture components indicates its chemical heterogeneity, expressed as periodic (with a period of 1.5 – 2.0 μm) composition oscillations of the intermetallic compound (Fig. 4, d) The deviation from the average stoichiometric ratio of components reaches up to 7 %.

Parameters of shock-wave compression of powder mixture

Параметры ударно-волнового сжатия порошковой смеси

Charge height, mm	Powder layer height, mm	Gasket thickness, mm	Detonation velocity of explosive, km/s	Powder heating temperature, °C	Pressure in reflected waves, GPa
70	7.0	1.5	4.2	777	11.5
85				831	12.5

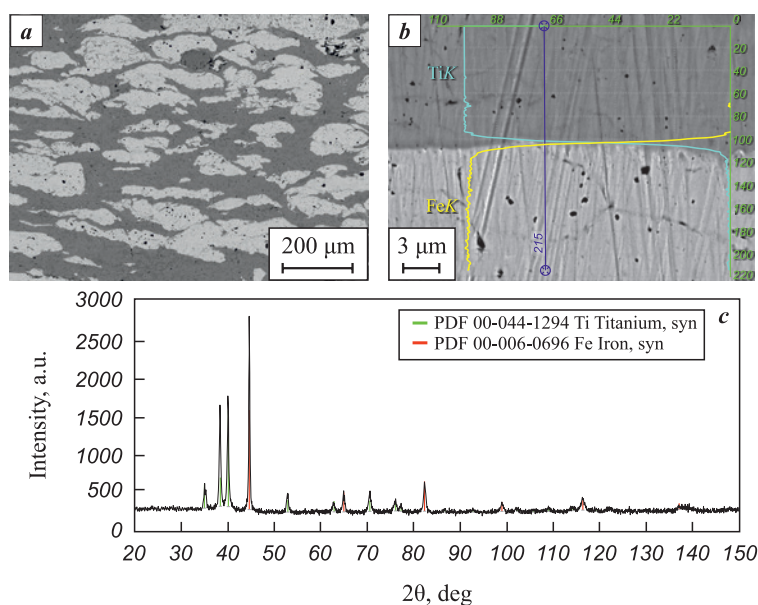


Fig. 3. Structure (a), chemical composition (b), and phase composition (c) of materials obtained under loading conditions: $t = 777\text{ }^{\circ}\text{C}$, $P = 11.5\text{ GPa}$

Рис. 3. Структура (a), химический (b) и фазовый (c) составы материалов, полученных в режиме нагружения: $t = 777\text{ }^{\circ}\text{C}$, $P = 11,5\text{ ГПа}$

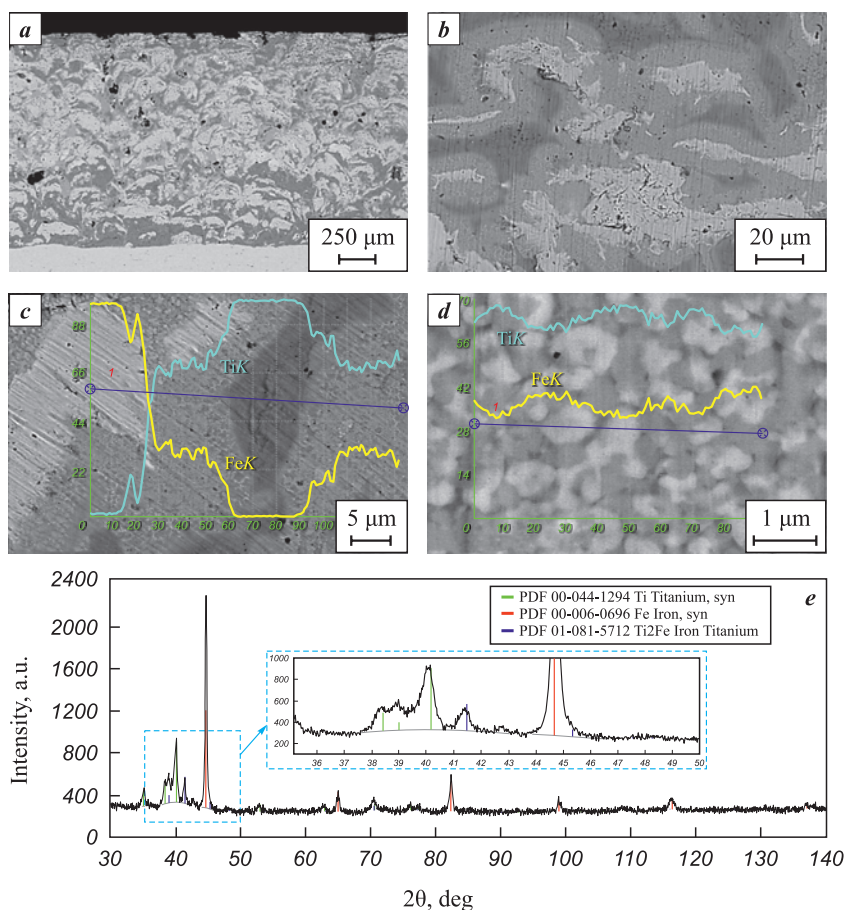


Fig. 4. Structure (a, b), chemical composition (c, d) and phase (e) composition of materials obtained under loading conditions: $t = 831\text{ }^{\circ}\text{C}$, $P = 12.5\text{ GPa}$

Рис. 4. Структура (a, b), химический (c, d) и фазовый (e) составы материалов, полученных в режиме нагружения: $t = 831\text{ }^{\circ}\text{C}$, $P = 12,5\text{ ГПа}$

Results and discussion

The data obtained from the conducted studies indicate that the mechanism of plastic deformation of titanium and iron particles during explosive compaction significantly influences the process of forming the metastable phase Ti_2Fe . Several hypotheses can be suggested about the nature of this influence. The most plausible hypothesis is that the intermetallic compound forms when a liquid phase is generated in the material during shock-wave treatment. This assertion is primarily supported by the experience of explosive welding of titanium alloys and steels [28; 29; 36].

Given that the contact melting temperature in the Ti–Fe system is low, at 1085 °C according to the phase diagram (see Fig. 1), achieving this temperature at particle boundaries during explosive compaction under conditions ensuring an average calculated heating temperature of 831 °C, combined with jet flows of metal and extreme temperature field heterogeneity, is quite likely. The heterogeneity of the formed intermetallic layer in this case could be a result of the simultaneous growth of its grains from a considerable number of crystallization centers within the liquid phase under rapid cooling due to heat dissipation into the “cold” areas of the structure and the metal substrate.

To test the proposed hypothesis, compacts obtained under loading conditions ensuring uniform plastic flow of the material particles without intermetallic phases were heated to 1100 °C in a vacuum with a holding time of 1 h, followed by cooling in an argon flow. As a result, the initial components of the compacts fully reacted with each other, forming a structure consisting of the intermetallic compounds $TiFe$ and Ti_2Fe (Fig. 5, *a, b*).

The associated impurity elements – carbon, oxygen, and nitrogen – were bound in titanium oxycarbonitrides (Fig. 5, *c*). Through coagulation and coalescence in the liquid phase, these formed relatively large inclusions that were uniformly distributed throughout the material (Fig. 5, *a, b*). The incorporation of part of the Ti into these inclusions appears to be the main reason why the $TiFe$ intermetallic phase formed in addition to the Ti_2Fe phase in the material’s structure. Addressing this issue requires increasing the Ti content (above stoichiometric) in the initial powder mixture.

Another factor likely contributing to the presence of the $TiFe$ intermetallic phase in the structure of compacts obtained by heat treatment is the significant increase in the duration of the interfacial interaction process when transitioning from purely explosive treatment to a combination of explosive and heat treat-

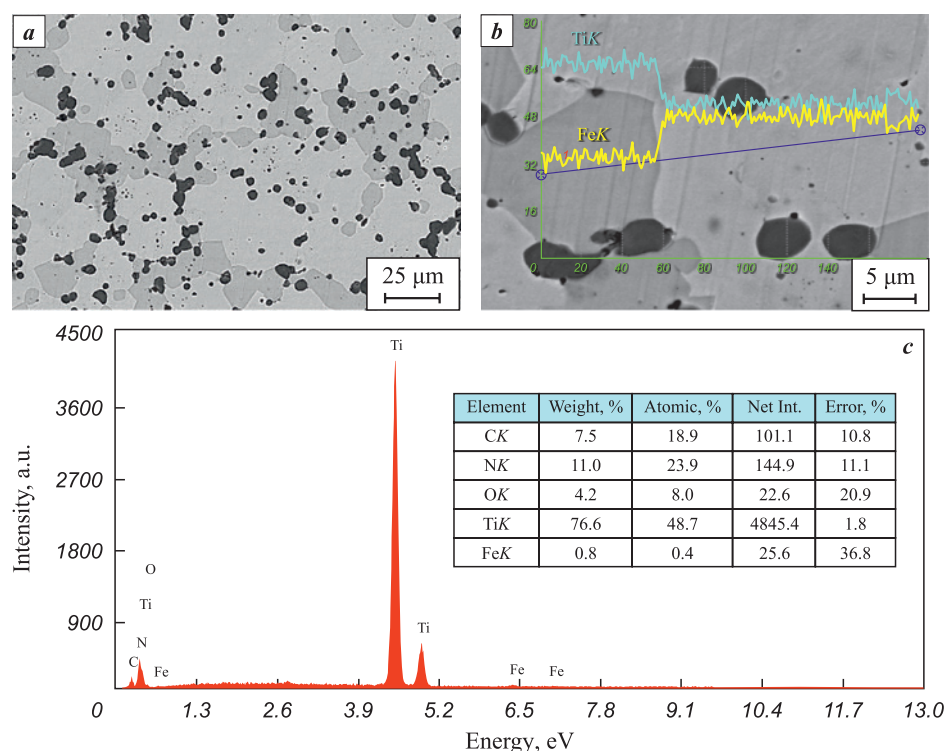


Fig. 5. Structure (*a*), chemical composition of intermetallic phases (*b*) and oxycarbonitride inclusions (*c*) of materials obtained by heat treatment of compacts

Рис. 5. Структура (*a*), химический состав интерметаллидных фаз (*b*) и оксикарбонитридных включений (*c*) материалов, полученных термической обработкой прессовок

ments. As a result, the likelihood of forming the stable TiFe phase and dissolving the metastable Ti_2Fe phase increased.

Conclusions

1. Under conditions of explosive compaction of iron and titanium powder mixtures that ensure uniform deformation of the initial particles without jet flows, the powder mixture compacts to an almost non-porous state while maintaining the original phase composition.

2. Using compaction regimes with localized plastic deformation and jet flows of the material particles in the original powder mixture forms a chemically heterogeneous metastable intermetallic Ti_2Fe as continuous layers between iron and titanium particles.

3. An effective method for producing materials with a high content of the metastable intermetallic Ti_2Fe is to combine explosive compaction of Fe and Ti powder mixtures with subsequent heat treatment in the intercritical temperature range (reactive sintering in the presence of a liquid phase).

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Кузьмин Е.В., Королёв М.П., Лысак В.И., Кузьмин С.В., Зарубин М.С., Петрушкин П.А., Львов В.А. Особенности формирования соединения композита титан–сталь при сварке взрывом с воздействием ультразвука. *Известия ВолгГТУ. Сер. Сварка взрывом и свойства сварных соединений*. 2020; 11(246):19–23.
<https://doi.org/10.35211/1990-5297-2020-11-246-19-23>

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S. V. Kuzmin – carried out calculations of explosive compaction parameters, participated in the discussion of the results.

V. I. Lysak – determined the purpose of the work, participated in the discussion of the results.

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Received 25.10.2023

Revised 10.04.2024

Accepted 15.04.2024

Статья поступила 25.10.2023 г.

Доработана 10.04.2024 г.

Принята к публикации 15.04.2024 г.