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RESEARCH

Structural and phase transformations in titanium alloys induced by ferrosilicon alloying

Oksana Baranovska[1*†], Gennadii Bagliuk[1], Olena Olifan[1], Seghii Korichev[1], Yaroslav Sytnyk[1] and Ahanov Andrii[2]

[1]Frantsevich Institute for Problems of Materials Science NASU, Ukraine, Kyiv
[2]Technical Center of the National Academy of Science of Ukraine, Ukraine, Kyiv

***Correspondence:**
Oksana Baranovska,
o.baranovska@ipms.kyiv.ua

[†]**ORCID:**
Oksana Baranovska
0000-0002-5023-0715

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The aim of this study was to investigate the structural and phase transformations in titanium alloys induced by ferrosilicon alloying and to enhance the physical and mechanical properties of titanium-based composite materials. These findings demonstrate that the use of FS65 ferroalloy as an alloying addition leads to active interaction with titanium hydride, resulting in the formation of a complex heterophase system. Using FS65 ferroalloy, boron carbide (B_4C), and carbon (C) as alloying additions, the research identifies the optimal synthesis temperature and examines the resulting microstructures and phase formations. The optimal synthesis temperature was found to be 1250°C. At this temperature, the phases TiC, TiSi, FeTi, and Ti_3SiC_2 were observed in the system 65 TiH_2 – 30 FeSi – 5 C (wt.%), while TiC, Ti_5Si_4, Fe_2Ti, and TiB_2 were identified in the system 65 TiH_2 – 30 FeSi – 5 B_4C (wt.%). The addition of 5% B_4C resulted in a finer microstructure with grain sizes ranging from 0.5 to 5 μm, compared to grain sizes of 5–10 μm with the addition of 5% C. The presence of B_4C also promoted the formation of TiB_2. The synthesized compacts, characterized by a fine-pored, spongy structure, are easily crushed, making them suitable for use as dispersed fillers in composite materials.

Keywords: titanium hydride, ferrosilicon, boron carbide, powder, thermal synthesis, microstructure, composite materials, intermetallic compound

1 Introduction

Titanium-matrix composites (TMCs) are known for their low specific gravity and corrosion resistance, making them advantageous materials. However, their high cost and reactivity with atmospheric gases, especially at high temperatures, limit their use in industries such as automobile and aircraft construction (1). To address these limitations, significant attention has been given to optimizing the alloying of titanium alloys to enhance their strength and high-temperature resistance while maintaining sufficient plasticity, as noted in various scientific studies. One effective method to achieve increased structural strength and heat resistance in titanium alloys involves the use of multicomponent alloying, as noted in sources (2–4).

Several factors are considered when choosing reinforcing compounds of a composite material (5). Notably, the material of these compounds should be characterized by a modulus of elasticity of the first kind, which significantly exceeds the Young's modulus of the matrix material (titanium), thus contributing to an increase in the hardness and strength of the composite.

Cubic β-Ti alloys are valuable biomedical alloys due to their low Young's modulus (E, which ranges from 40 to 70 GPa), high specific strength and plasticity, and good corrosion resistance (6, 7). However, they are metastable and

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prone to forming secondary phases, such as α and ω, which increase the E value to approximately 110 GPa (8–11).

Due to their metastable nature, these alloys require β-stabilizers to achieve high structural stability. The largest group of industrial titanium alloys is represented by two-phase (α++β) alloys, which are distinguished by increased strength, sufficient plasticity, and good workability through pressure processing methods (3). These alloys are produced using β-stabilizing alloying elements that provide the ability for solid solution or thermal strengthening necessary to achieve the desired properties.

The material of the strengthening particles should be characterized by stability within the titanium matrix and should not react with it during operation. The structural stability of β-Ti alloys is usually evaluated using the Mo equivalent (Mo_{eq}) (12). According to (12), Fe, Cr, Cu, Ni, Co, and Mn undergo eutectoid reactions with Ti. Their (Mo_{eq}) coefficients exceed 1.0, indicating a greater β-stabilizing ability compared to monotectoid elements like Mo, W, and V.

The application of complex ferroalloys in the alloying of titanium-based powder mixtures can address the challenge of enhancing the physical and mechanical properties of the resulting products while reducing the cost of the final product. Ferroalloys, especially those composed of a complex of different alloying metals, are very brittle and do not require significant expenditures for their grinding into a powder state. The granulometric composition of powder mixtures is easily regulated within certain limits, allowing for good compaction of the powder mixtures during forming and minimal shrinkage during sintering.

A significant increase in the strength characteristics of titanium alloys is also achieved due to dispersion strengthening with the help of carbides (TiC, SiC, B_4C), borides (TiB_2, TiB), oxides (Al_2O_3, ZrO_2, TiO_2, R_2O_3, where R is a heavy earth element), intermetallic compounds (Ti_3Al, TiAl), and Ti_5Si_3 (13). In several studies (14–16), it is shown that among these additives, titanium silicide (Ti_5Si_3) is the most attractive due to its low density (4.26 g/cm^3), high melting point (2130°C), high oxidation resistance due to the formation of a stable surface layer of SiO_2, and the ability to maintain high strength up to 1200°C.

Previous studies (17, 18) have investigated the influence of ferrosilicomanganese and demonstrated that its addition significantly enhances the mechanical properties and structural stability of titanium alloys.

Considering the data, based on the analysis of the structural stability of β-Ti, and the economic feasibility and availability, it is interesting to explore the possibility of using complex ligatures for alloying sintered titanium alloys that contain such inexpensive and widely available β-stabilizing elements such as iron, manganese, and silicon. Additionally, ferroalloys have lower melting temperatures, which simplifies the alloying process.

Alloying titanium with ferrosilicon significantly improves its mechanical properties, such as strength, wear resistance, and hardness, making it more suitable for applications under high loads and in aggressive environments. Silicon, which is part of ferrosilicon, enhances the corrosion resistance of titanium alloys, especially in acidic and saline environments, which is crucial for use in marine, chemical, and aerospace industries. Furthermore, ferrosilicon is a relatively inexpensive and accessible alloying element, which can reduce the cost of material production. Due to its unique properties, ferrosilicon enables the creation of multifunctional titanium alloys that possess high strength, corrosion resistance, and good thermal conductivity simultaneously.

Ferrosilicon is an alloy of silicon and iron, used as a deoxidizer and alloying additive in steel production. Ferrosilicon includes a large group of alloys in the Fe–Si system. Ferrosilicon obtained in electric furnaces can contain 19–92% silicon and forms a series of silicides—FeSi, Fe_2Si_3, $FeSi_2$, Fe_3Si_2, and others, with FeSi being the most robust, having a melting point of 1410°C.

The use of ferrosilicon for alloying titanium is interesting because its addition promotes the formation of chemical compounds such as TixMy, which undergo eutectoid transformation, thereby improving the mechanical properties of the material, reducing production costs, and facilitating the processing, opening prospects for the application of these alloys in various industries, including aerospace, automotive, and medical fields.

Optimal compositions of ferrosilicon include alloys based on singular phases formed in the solid state, which are stable phases in the homogeneity region of solid and liquid solutions. According to ISO 5445-80, optimal ferrosilicon alloys include FS20 and FS65, which are formed based on singular iron silicides.

It is important to note that alloying titanium with ferroalloys, particularly ferrosilicon, has not been previously explored in the scientific literature. This study is the first to investigate this approach, which could significantly impact the development of titanium composite materials by increasing their mechanical properties while simultaneously reducing production costs.

On this basis, this study aims to investigate the effect of ferrosilicon alloying on the structural and phase transformations in titanium alloys, with a focus on understanding its influence on microstructural evolution.

2 Methodology

As the base of charge, we used powders of PTKh-80 titanium hydride with particle sizes $\leq$ 63 μm. As an alloying element capable of providing high-performance composite material, the FS65 ferroalloy (particle size $\leq$ 80 μm), composed of Si = 66.4%; Fe 32.26%; Mn = 0.22%; *S* = 0.003%;

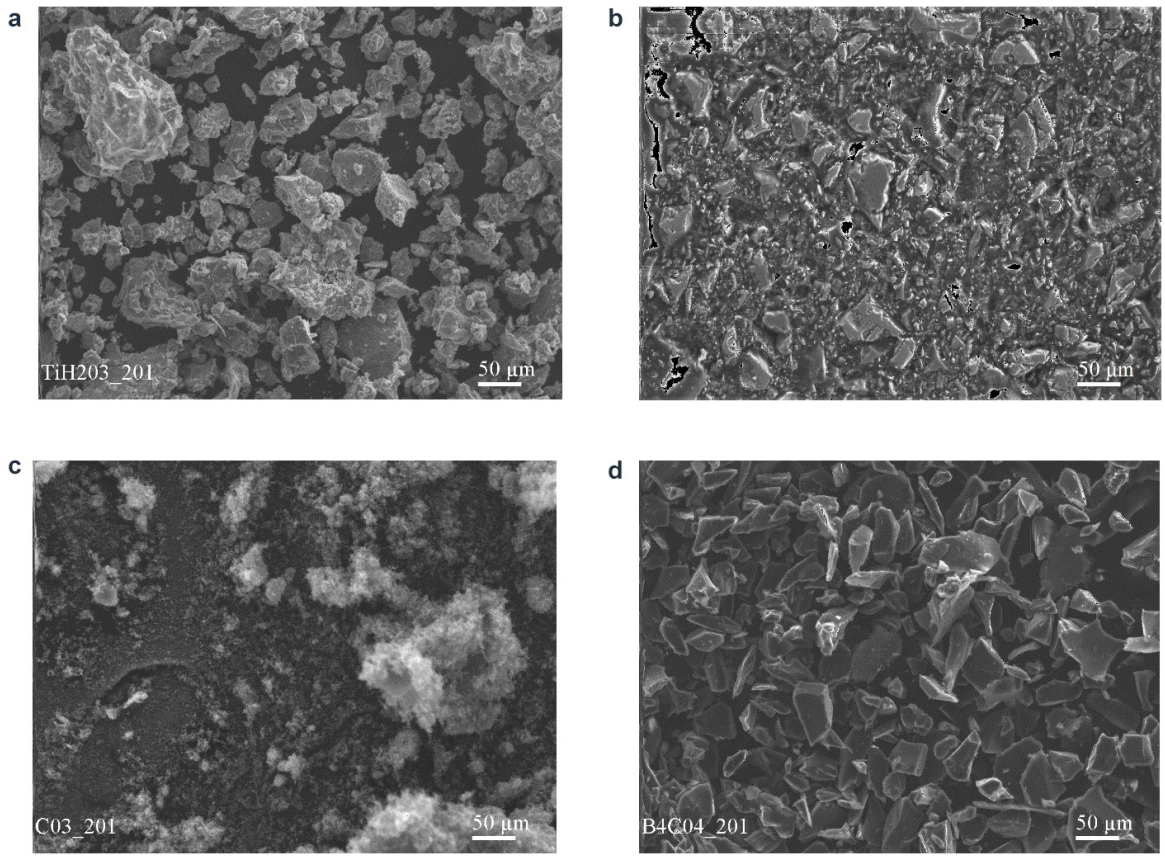

FIGURE 1 | Morphology of the input powders of titanium hydride **(a)**, ferrosilicon **(b)**, carbon **(c)**, and boron carbide **(d)**.

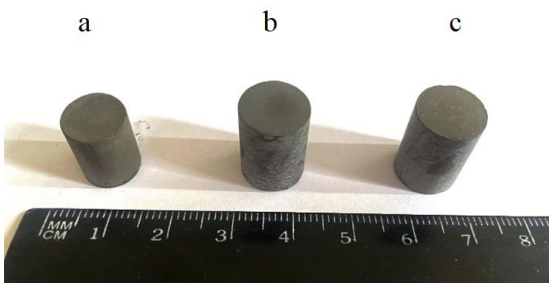

FIGURE 2 | External appearance of the samples after thermal synthesis of green compact **(a)**, 65 TiH_2 – 30 FeSi – 5 C (wt.%) **(b)** and 65 TiH_2 – 30 FeSi – 5 B_4C (wt.%) **(c)** at 1250°C.

P = 0.03%; Cr = 0.17%; and Al = 0.92% (wt.%), was selected. Boron carbide and carbon were used as refractory compounds. The initial powder morphology of titanium hydride, ferrosilicon, carbon, and boron carbide is shown in Figure 1a–d, respectively. Considering that iron and silicon form an unlimited series of solid solutions and have excellent wettability with respect to titanium, this promotes better homogeneity and bonding of components in the alloy. The initial mixture components were mechanically activated in a planetary mill in a medium of alcohol for 10 min with grinding bodies in the form of balls made of ShCh15 steel with diameters of 4–12 mm. The ratio of the masses of balls and powder was equal to 6:1. The resulting mixture was dried in air and then pressed into briquettes under a pressure of 350 MPa for the 65 TiH_2 – 30 FeSi – 5 C (wt.%) mixture and 400 MPa for the 65 TiH_2 – 30 FeSi – 5 B_4C (wt.%) mixture. It is important to note that compression pressures ranging from 200 MPa to 500 MPa were tested. It was found that only at pressures of 350 MPa (for the 65 TiH_2 – 30 FeSi – 5 C (wt.%) mixture) and 400 MPa (the 65 TiH_2 – 30 FeSi – 5 B4C (wt.%) mixture) did the material maintain its integrity without delamination. Then we sintered the specimens in a vacuum furnace of the SShVL-1.2,5/25-M04 type with a heating rate of 5–7°C/min in two consecutive stages: heating to 600°Ñ with isothermal holding for 30 min (to remove the main part of hydrogen) and heating to 1250°Ñ with holding for 60 min.

The micrographs of initial powders and sintered sample surfaces were investigated by an XJL-17AT optical microscope and a JEOL Superprobe 733 scanning electron microscope. The phase composition of the master alloy was X-ray analyzed using a DRON-3 diffractometer with a Cu-Kα-radiation source in the range of 20 ÷ 90° angles with step-by-step scanning. The specimen rotated around its axis during the diffraction. The correspondence of the diffraction lines was carried out using the PDF-2 database.

Differential thermal analysis (DTA) was conducted using a setup designed and manufactured at the Department

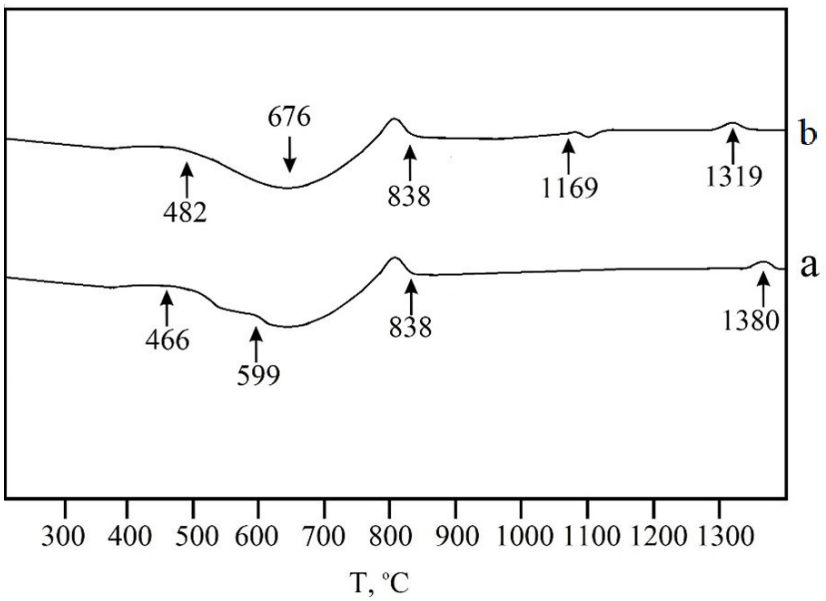

FIGURE 3 | Differential thermal analysis of powder mixtures: 65 TiH_2 – 30 FeSi – 5 C (wt.%) **(a)**, 65 TiH_2 – 30 FeSi – 5 B_4C (wt.%) **(b)**.

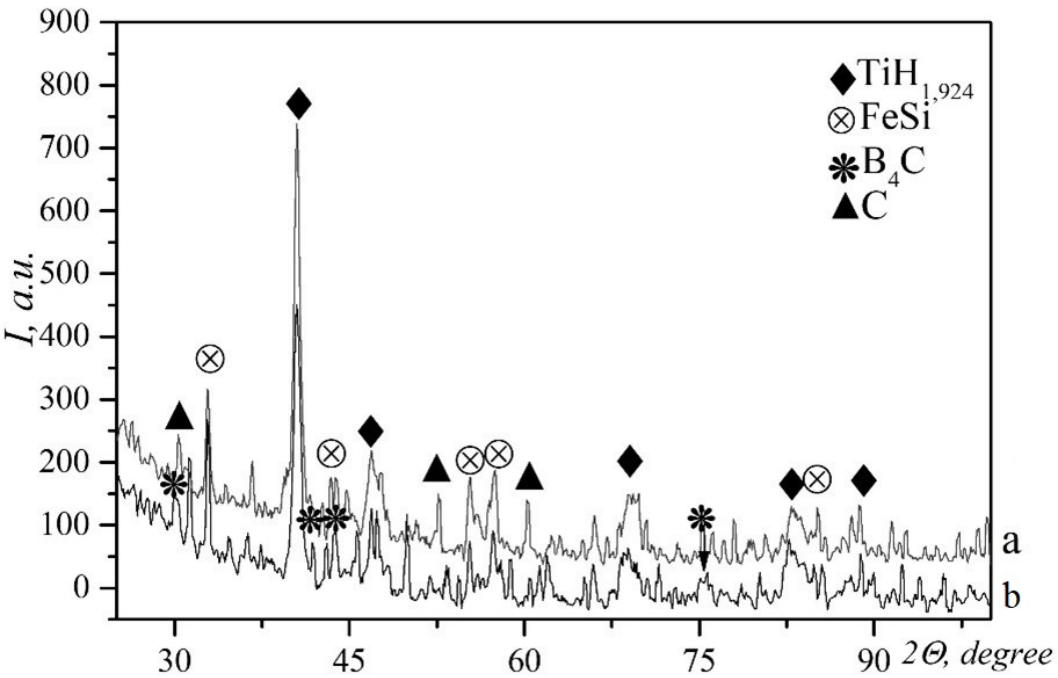

FIGURE 4 | X-ray phase analysis of initial powder mixtures: 65 TiH_2 – 30 FeSi – 5 C (wt.%) **(a)**, 65 TiH_2 – 30 FeSi – 5 B_4C (wt.%) **(b)**.

of Physical Chemistry of Inorganic Materials, Frantsevich Institute for Problems of Materials Science of NAS of Ukraine (IPM NANU). The sensor used consists of a molybdenum block on a tungsten leg and tungsten/VR20 wire thermocouples. High-purity helium at a pressure of 100 kPa was used as a protective environment, and the heating and cooling rates were generally 20°C/min. Samples were placed in ceramic crucibles made of Al_2O_3. Thermocouples were calibrated using a set of primary and secondary reference materials MPTSh-90: Al, Ag, Au, Pd, Pt, Rh, Ru, and auxiliary (Fe and Si). The reproducibility of the international practical temperature scale (IPTS), which is a combination of random and instrumental errors, ranges from 3 to 10°C in the interval of 1200-1800°C.

3 Results

Experimental synthesis results show that at a temperature of 1000°C, the samples do not sinter and represent a

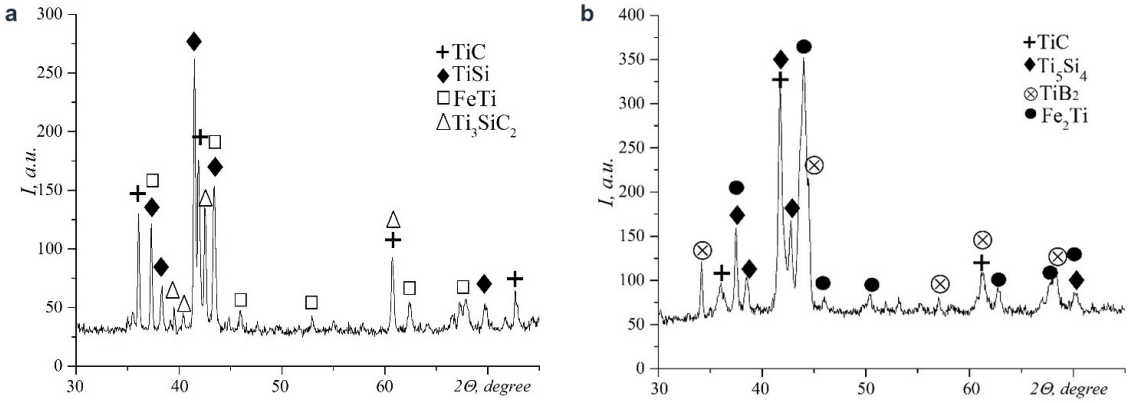

FIGURE 5 | Fragments of X-ray diffraction patterns of alloys of 65 TiH_2 – 30 FeSi – 5 C (wt.%) **(a)** and 65 TiH_2 – 30 FeSi – 5 B_4C (wt.%) **(b)** systems after thermal synthesis at 1250°C.

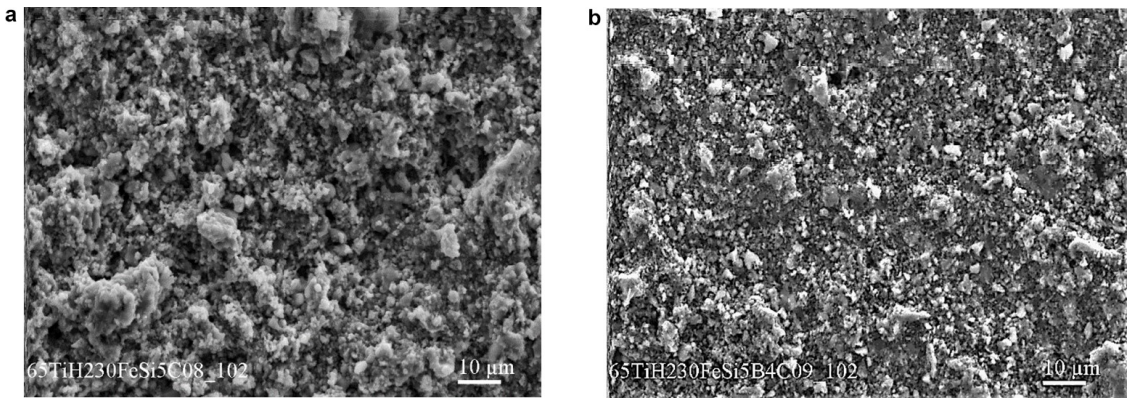

FIGURE 6 | SEM images of microstructure of 65 TiH_2 – 30 FeSi – 5 C (wt.%) **(a)** and 65 TiH_2 – 30 FeSi – 5 B_4C (wt.%) **(b)** systems synthesized at 1250°C.

loose sponge with weak interparticle bonds. At 1000°C, the necessary diffusion activity, which ensures the formation of strong interparticle bonds, is not achieved. Therefore, the material particles remain insufficiently bonded, forming a sponge-like structure with large pores. Increasing the synthesis temperature to 1250°C leads to an increase in the volume of synthesized samples. The sintered samples are strong, porous (P_1 = 25.36%, P_2 = 26.16%) conglomerates, resembling ceramics in appearance (Figure 2).

DTA for two powder mixtures (Figure 3) is almost identical and shows that at temperatures up to 600°C, desorption of hydrogen from titanium hydride powder occurs. Increasing the temperature to approximately 820°C leads to an exothermic reaction because of the interaction between Fe and Ti. According to the phase diagram of the Fe–Ti system (19), the formation of three eutectics is possible. The lowest of these forms is at 1100°C between titanium and FeTi. The authors of the study (20) showed that mechanical activation for 5–10 min affects the temperature of the interaction between Ti and Fe. The eutectic formation temperature in activated mixtures is somewhat lower than 1100°C.

The nature of the exothermic effect at 1169°C for the 65 TiH_2 – 30 FeSi – 5 B_4C system (Figure 3b) may be associated with the interaction of Ti with B_4C. For the 65 TiH_2 – 30 FeSi – C system (Figure 3a), the exothermic peak at 1169°C is not observed. The endothermic peak observed after the exothermic one at 1169°C can be explained by the process of transformation $TiC_{0.5}$? $TiC_{1.0}$, which, according to (21), occurs due to the simultaneous dissolution of non-stoichiometric carbides in the melt and their recrystallization into carbides with a higher carbon concentration.

X-ray phase analysis of the initial mixtures showed (Figure 4) that the initial titanium hydride corresponds to the formula $TiH_{1.924}$. Ferrosilicon FS-65 consists of a cubic iron silicide compound, FeSi. The initial boron carbide corresponds to the formula B_4C, and the soot corresponds to carbon.

The diffractograms of mixtures synthesized at 1250°C (Figure 5) showed the presence of TiC, TiSi, FeTi, Ti_3SiC_2 phases for the 65 TiH_2 – 30 FeSi – 5 C (wt.%) system and TiC, Ti_5Si_4, Fe_2Ti, TiB_2 phases for the 65 TiH_2 – 30 FeSi – 5 B_4C (wt.%) system, respectively.

Incorporating 5% C results in X-ray peaks indicating the presence of the FeTi intermetallic compound (Figure 5a),

while the addition of B_4C shows the intermetallic phase as the Fe_2Ti compound (Figure 5b). The formation of the FeTi phase initially stems from mutual diffusion between Fe and Ti, with Fe diffusing into Ti much faster than Ti into Fe at the same temperature (22). This rapid diffusion leads to increased Fe concentration in the melt and subsequent crystallization of Fe_2Ti.

X-ray diffractograms of both synthesized compacts also revealed the presence of silicide phases: TiSi for the 65 TiH_2 – 30 FeSi – 5 C (wt.%) system and Ti_5Si_4 for the 65 TiH_2 – 30 FeSi – 5 B_4C (wt.%) system. The formation of these phases occurs according to reactions (1 and 2) and is thermodynamically favorable since the corresponding reactions lead to a significant reduction in Gibbs energy:

$$Ti + Si \rightarrow TiSi \quad (1)$$

$$5Ti + 4Si \rightarrow Ti_5Si_4 \quad (2)$$

In the study (23), Gibbs energies at 1200°C were determined. Thus, for the formation of TiSi, the change in Gibbs energy $\Delta G = -71.4$ kJ/mol, and for Ti_5Si_4, $\Delta = -74.8$ kJ/mol. This indicates that these reactions are very favorable from an energy point of view. Also, during the synthesis process, carbon (C) diffuses into titanium, forming titanium carbide (TiC) peaks on the diffractogram (Figure 5a). As the temperature increases, the formed TiC interacts with Ti_5Si_3, leading to the formation of the more complex phase Ti_3SiC_2, whose peaks are indexed on the X-ray diffraction pattern.

The formation of FeB in the 65 TiH_2 – 30 FeSi – 5 B_4C mixture was not detected (Figure 5b) because the free energy of the formation of Ti-B phases is significantly higher than that of Fe-B phases. During heating, carbon (C) and boron (B) atoms dissolve in the Fe–Ti melt, forming a homogeneous Fe–Ti–C–B melt. When the concentration of C and B atoms reaches a critical level, titanium reacts with these elements, leading to the formation of TiC and titanium diboride (TiB_2) in the liquid phase, accompanied by the release of a significant amount of thermal energy. Thus, the presence of these reactions explains the absence of FeB formation and indicates the predominance of reactions involving titanium.

It was not possible to determine the presence of cementite or other phases and compounds.

The microstructure of synthesized compacts is characterized by uniformly distributed carbide and boride phases. The structure using 5% B_4C is more finely dispersed with a grain size of 0.5–5 μm (Figure 6b), whereas the grain size with 5% C is 5–10 μm (Figure 6a). In addition, the microstructure of the materials contains conglomerates of grains, which may arise due to prior mechanical activation of the powder mixture. During mechanical activation, the powder undergoes intense plastic deformation, leading to distortion of the alloy's crystal lattice. This distortion can result in the formation of small crystallites, which subsequently combine into conglomerates.

The resulting compacts represent a fine-dispersed porous sponge that is easily crushed, allowing these materials to be used as a master alloy for improving the wear resistance of titanium alloys (24, 25), as well as a filler for epoxy polymers to enhance their physical, mechanical, and adhesive properties (26, 27).

4 Conclusion

(1) The use of ferroalloys as an alloying additive leads to their active interaction with titanium hydride. This interaction is accompanied by the dissociation of the master alloys, resulting in the formation of a complex heterophase system, with subsequent phases forming: TiC, TiSi, FeTi, and Ti_3SiC_2 for the 65 TiH_2 – 30 FeSi – 5 C (wt.%) system, and TiC, Ti_5Si_4, Fe_2Ti, and TiB_2 for the 65 TiH_2 – 30 FeSi – 5 B_4C (wt.%) system. The presence of B_4C leads to the formation of TiB_2.

(2) Adding 5% B_4C results in a finer microstructure with grain sizes ranging from 0.5 to 5 μm, whereas the grain size with the addition of 5% C is 5–10 μ m.

(3) These materials can be used as master alloys to improve the wear resistance of titanium alloys and can also serve as fillers for epoxy polymers.

Authors' contributions

OB led the research project, designed the experiments, conducted the experimental work, and wrote the entire manuscript. GB provided essential supervision, guidance, and support throughout the research project, including the conceptualization of the study. OO and SK were responsible for performing the X-ray diffraction analyses and interpreting the resulting data, which were crucial for the phase composition determination. YS handled all technical tasks, including sample preparation, pressing, and cutting, ensuring the accuracy and consistency of the experimental samples. AA conducted the microstructural analysis using advanced microscopy techniques, producing detailed images that were critical for understanding the material's structural properties. All authors reviewed and approved the final version of the manuscript and agreed to be accountable for all aspects of the work.

Conflict of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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