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## Influence of Zr and V dopants on the degree of the $\text{Ti}_3\text{SiC}_2$ MAX phase formation by mechanochemistry

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As a result of mechanically activated self-propagating synthesis, from the initial mixture of Ti, Si, and C powders or a mixture of Ti, Si, and C powders with the addition of 5 wt. % Zr or with the addition of 5 wt. % V, the MAX phase  $\text{Ti}_3\text{SiC}_2$  was obtained. The effect of doping the MAX phase  $\text{Ti}_3\text{SiC}_2$  with zirconium and vanadium on the degree of its formation was investigated. It was found that doping the MAX phase  $\text{Ti}_3\text{SiC}_2$  with 5 wt. % V led to an increase in its amount and, accordingly, a decrease in the amount of the secondary TiC phase in the final product. When doping the MAX phase with 5 wt. % Zr, on the contrary, its amount decreased, and the amount of the TiC phase increased.

**Keywords:** MAX phases,  $\text{Ti}_3\text{SiC}_2$ , mechano-chemical method, unit cell.

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### Introduction

Layered ternary carbides (MAX phases) are of great interest as a promising material for high-temperature ceramics, electrical contacts, sensors and protective coatings owing to the unique combination of properties characteristic of both metals and ceramics [1].

At the same time, MAX phases effectively enhance the hydrogen sorption properties and the onset temperature of decomposition of magnesium hydride [2 - 5]. It has been shown [6 - 10] that a small amount of MAX phase additive (5 - 7 wt.%) to  $\text{MgH}_2$  (or Mg), similarly to the addition of transition metals or their compounds, leads to an improvement in the kinetics of magnesium hydrogenation and  $\text{MgH}_2$  dehydrogenation.

$\text{Ti}_3\text{SiC}_2$  MAX phase is also a precursor for obtaining MXene  $\text{Ti}_3\text{C}_2$ , which, in turn, due to its high electrical conductivity, ability to accept ions and provide fast ion transport, has prospects for utilization as an electrode material for batteries [11] and supercapacitors [12]. Today, MXenes have already shown their potential in many other fields, such as ecology, biomedicine and catalysis [11 - 14]. One of the pathways to obtain  $\text{Ti}_3\text{C}_2$  MXene is the etching of silicon from  $\text{Ti}_3\text{SiC}_2$  [15].

The synthesis of the  $\text{Ti}_3\text{SiC}_2$  MAX phase is mainly

carried out by hot isostatic pressing (HIP), spark plasma sintering (SPS) and self-propagating high-temperature synthesis (SHS) [16]. Common problems of these methods are the use of high temperatures, the availability of complex and expensive equipment for the synthesis of high-purity MAX phase  $\text{Ti}_3\text{SiC}_2$  and its low yield. Therefore, high-energy milling of the initial components in a planetary mill was applied as a prospective synthesis route for the preparation of  $\text{Ti}_3\text{SiC}_2$  MAX phase powder [17 - 19].

The aim of this work was to study the effect of doping the  $\text{Ti}_3\text{SiC}_2$  MAX phase with zirconium and vanadium on the degree of its formation by the method of mechanochemistry, i.e. to investigate the possibility of reducing the amount of the secondary TiC phase by doping with Zr and V as a result of synthesis.

To achieve the goal, in this work, the  $\text{Ti}_3\text{SiC}_2$  MAX phase was synthesized from the initial mixture of Ti, Si and C powders, as well as it was obtained with the addition of Zr powder (5 wt. %) or V powder (5 wt. %) to the initial mixture. Qualitative and quantitative X-ray phase analysis of the synthesized samples was conducted, the parameters of the unit cells were determined and the microstructure and morphology of the samples were investigated.

The choice of zirconium and vanadium as alloying

elements was due to the fact that doping the MAX phase  $\text{Ti}_3\text{SiC}_2$  with each of these transition metals, which are hydride-forming elements, could facilitate significant increase the degree of its formation and reduce the amount of secondary phases, for example, TiC.

## I. Materials and methods

As reactants in the present investigation were used titanium powder (Zaporizhzhya Titanium and Magnesium Plant), high-purity graphite OSCh-8-4 (GOST 23463-79), ( $S = 0.6 \text{ m}^2/\text{g}$ ), crystalline silicon ( $S = 0.5 \text{ m}^2/\text{g}$ ), zirconium powder PZrK, vanadium metal VnM-1. The milling was performed in argon atmosphere in a planetary mill AIR-015M at a disk and vial rotation speed of 735 rpm and 1840 rpm, respectively. The balls to powder weight ratio was 5:1. The amount of titanium introduced constituent 74 wt.%, silicon – 14 wt.%, carbon – 12 wt.%. When doping, 5 wt.% zirconium or vanadium was added to the mixture.

X-ray diffraction studies of the obtained MAX phase samples were carried out on a DRON-3M diffractometer with Bragg-Brentano focusing in monochromatic cobalt radiation with a graphite monochromator on diffracted beams. The X-ray intensity was measured over diffraction  $2\theta$  from  $38$  to  $72^\circ$  with a scanning step size of  $0.10^\circ$  and step time of 7 s at a mode of 30 kV, 12 mA. The crystal lattice parameters of the synthesized  $\text{Ti}_3\text{SiC}_2$  MAX phase sample, as well as  $\text{Ti}_3\text{SiC}_2$  with the addition of 5 wt. % Zr and  $\text{Ti}_3\text{SiC}_2$  with the addition of 5 wt. % V, mass fraction of phases (%) were determined by the full-profile Rietveld method using the fullprof Powder Cell 2.4 software (<https://powdercell-for-windows.com/2.4/>). Software. Informer. Com /2.4/).

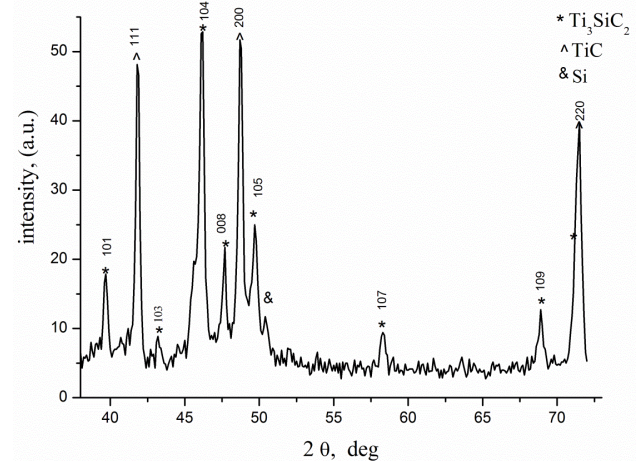
The particle size of the powder of MAX phases obtained by the mechanochemical method was investigated using scanning electron microscopy in the secondary electron mode at an accelerating voltage of 25 kV on a Super-Probe 733 equipment.

## II. Results and discussion

Fig. 1 shows the diffractogram of the synthesized sample, which was obtained from a mixture of the initial Ti, Si and C powders. This sample revealed to be two-phase: the diffractogram contains reflections from the  $\{101\}$ ,  $\{103\}$ ,  $\{104\}$ ,  $\{008\}$ ,  $\{105\}$  and  $\{109\}$  planes of

the hexagonal  $\text{Ti}_3\text{SiC}_2$  phase of the space group S.G.:  $P6_3/mmc(194)$  with unit cell parameters  $a = 3.0574 \text{ \AA}$ ;  $c = 17.6283 \text{ \AA}$ ,  $V = 142.708 \text{ \AA}^3$  and reflections from the  $\{111\}$ ,  $\{200\}$ ,  $\{220\}$  planes of the cubic phase of TiC of the space group S.G.:  $Fm\bar{3}m(225)$  with the lattice period  $a = 4.3333 \text{ \AA}$  and  $V = 81.368 \text{ \AA}^3$ . The diffraction pattern also contains a reflection from the  $\{311\}$  plane of orthorhombic silicon. The experimentally determined parameters and volumes of the crystal lattices of the synthesized phases are summarized in Table 1.

As a result of quantitative analysis by the full-profile Rietveld method, it was found that the sample contains 55 wt. % of the  $\text{Ti}_3\text{SiC}_2$  phase and 45 wt. % of the TiC phase.



**Fig. 1.** Diffractogram of a sample synthesized from a mixture of Ti, Si and C powders.

The diffraction pattern from the sample with the synthesized MAX phase  $\text{Ti}_3\text{SiC}_2$ , doped with 5 wt. % Zr, is shown in Fig. 2. This sample also turned out to be two-phase: phase analysis showed the presence of the  $\text{Ti}_3\text{SiC}_2$  phase with a lattice period  $a = 3.0551 \text{ \AA}$ ,  $c = 17.6525 \text{ \AA}$ ,  $V = 142.689 \text{ \AA}^3$  and the TiC phase with a period  $a = 4.3310 \text{ \AA}$  and a unit cell volume  $V = 81.250 \text{ \AA}^3$ . X-ray phase analysis revealed the presence of traces of Ti and orthorhombic silicon, while no pure zirconium was detected. Quantitative analysis revealed that this sample contains 32.3 wt. %  $\text{Ti}_3\text{SiC}_2$  and 67.7 wt. % TiC.

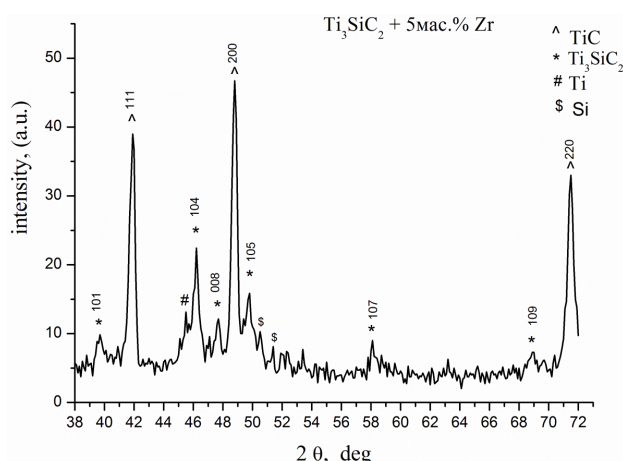
As can be seen from Fig. 2, doping with zirconium leads to a change in the peak intensities compared to the intensity of the same peaks in the diffractogram of the undoped  $\text{Ti}_3\text{SiC}_2$  sample (see Fig. 1) and, as quantitative analysis has shown, to a decrease in the amount of the

**Table 1.**

Crystal lattice parameters ( $a$ ,  $c$ – $\text{\AA}$ ) and unit cell volume ( $V$ – $\text{\AA}^3$ ) of the synthesized phases and their quantitative composition (%).

| Sample after synthesis without Zr & V dopant |              | Sample after synthesis with Zr dopant |               | Sample after synthesis with V dopant |              |
|----------------------------------------------|--------------|---------------------------------------|---------------|--------------------------------------|--------------|
| Phase $\text{Ti}_3\text{SiC}_2$              | Phase TiC    | Phase $\text{Ti}_3\text{SiC}_2$       | Phase TiC(Zr) | Phase $\text{Ti}_3\text{SiC}_2$ (V)  | Phase TiC(V) |
| $a = 3.0574$                                 | $a = 4.3333$ | $a = 3.0551$                          | $a = 4.3310$  | $a = 3.0496$                         | $a = 4.3268$ |
| $c = 17.6283$                                |              | $c = 17.6525$                         |               | $c = 17.5829$                        |              |
| $V = 142.708$                                | $V = 81.368$ | $V = 142.689$                         | $V = 81.250$  | $V = 141.615$                        | $V = 81.014$ |

$\text{Ti}_3\text{SiC}_2$  phase and an increase in the amount of the TiC phase. In our opinion, one possible explanation for this established fact could be the following. During the synthesis of the MAX phase  $\text{Ti}_3\text{SiC}_2$  by the mechanochemical method, zirconium, present in the mixture of Ti, Si and C powders, which has good catalytic properties of carbide formation, could contribute to a significantly greater improvement in the reaction rate of the formation of the TiC phase than that of the  $\text{Ti}_3\text{SiC}_2$  phase. It can also be assumed that in the process of synthesis of the MAX phase  $\text{Ti}_3\text{SiC}_2$  with the addition of 5 wt. % Zr, zirconium atoms enter the TiC lattice. As a result of such entry, the volume of the unit cell of the TiC phase ( $V = 81.250 \text{ \AA}^3$ ) decreased compared to the volume of the TiC cell without the addition of Zr ( $V = 81.368 \text{ \AA}^3$ ). At the same time, the volume of the unit cell of the MAX phase  $\text{Ti}_3\text{SiC}_2$  practically did not change:  $V = 142.708 \text{ \AA}^3$  before doping and  $V = 142.689 \text{ \AA}^3$  after doping with zirconium.

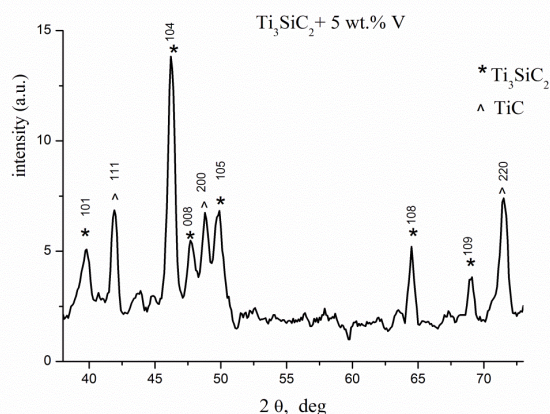


**Fig. 2.** Diffraction pattern of a sample synthesized from a mixture of Ti, Si and C powders with the addition of Zr powder (5 wt.%).

A completely different picture was observed when 5 wt. % of vanadium powder was added to the mixture of Ti, Si and C powders, and mechanochemical synthesis was carried out. The diffractogram of the MAX phase  $\text{Ti}_3\text{SiC}_2$  doped with 5 wt. % V is shown in Fig. 3. This sample revealed to be two-phase with the presence of the  $\text{Ti}_3\text{SiC}_2$  phase with a lattice period  $a = 3.0546 \text{ \AA}$ ,  $c = 17.6294 \text{ \AA}$  and with a unit cell volume of the phase  $V = 142.456 \text{ \AA}^3$  and the TiC phase with a lattice period  $a = 4.3268 \text{ \AA}$ ,  $V = 81.014 \text{ \AA}^3$  (see Table 1). The diffraction pattern does not contain reflections of pure vanadium, and no traces of titanium or silicon were detected.

From the analysis of the diffractogram shown in Fig. 3, it can be concluded that doping with vanadium, as in the case of zirconium, leads to a change in the peak intensities of the MAX phase  $\text{Ti}_3\text{SiC}_2$  doped with 5 wt. % V, in comparison with the diffractogram from the sample with the MAX phase  $\text{Ti}_3\text{SiC}_2$  synthesized only from a mixture of Ti, Si and C powders (see Fig. 1). Thus, the intensity of the lines from the {104} and {105} planes of the MAX phase  $\text{Ti}_3\text{SiC}_2$  significantly exceeds the intensity of the lines from the {111} and {200} planes of the TiC phase. Quantitative X-ray phase analysis showed a noticeable increase in the amount of the MAX phase

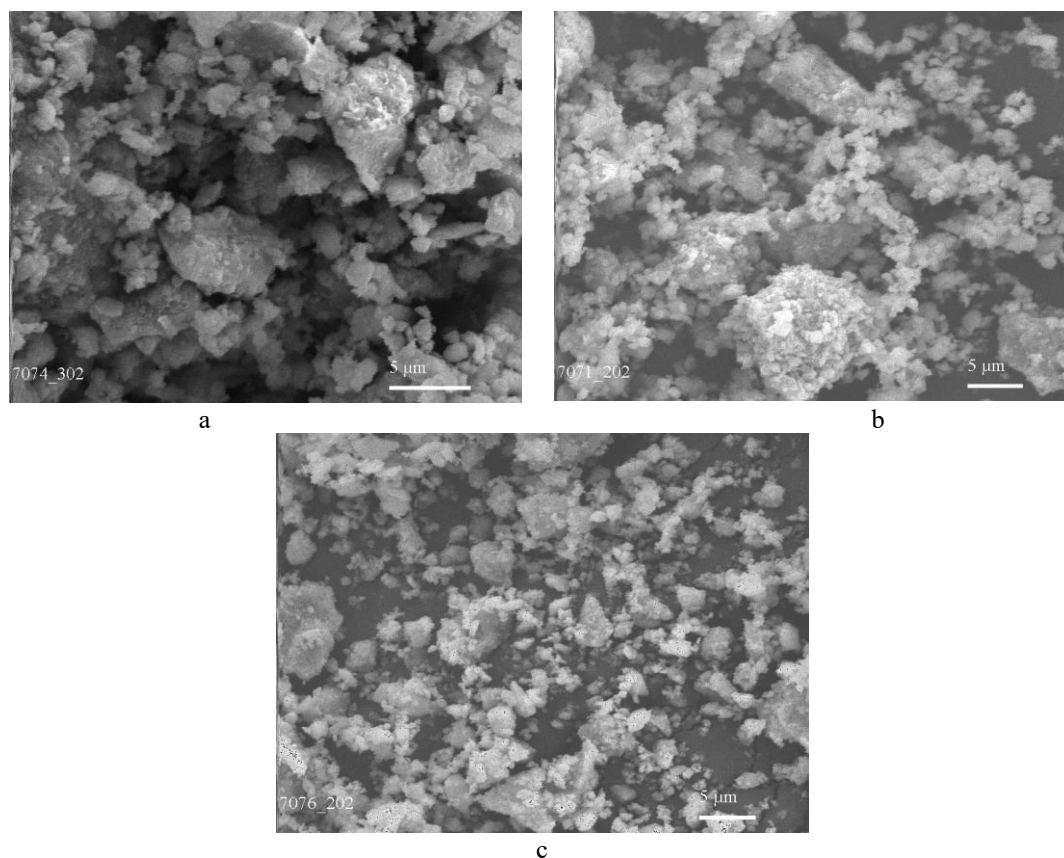
$\text{Ti}_3\text{SiC}_2$  from 55 wt.% to 74.4 wt. %, whereas the amount of the TiC phase decreases from 45 wt.% to 25.6 wt. %. The increase of the  $\text{Ti}_3\text{SiC}_2$  quantity, which is observed in the case of its doping with vanadium, in our opinion can be explained by the fact that vanadium, unlike zirconium, in the process of synthesis of the MAX phase  $\text{Ti}_3\text{SiC}_2$  (i.e. milling of a mixture of Ti, Si and C powders with the addition of 5 wt.% of vanadium powder) could contribute to a significant increase in the reaction rate of the formation of the  $\text{Ti}_3\text{SiC}_2$  phase, and not the TiC phase.



**Fig. 3.** Diffraction pattern of a sample synthesized from a mixture of Ti, Si and C powders with the addition of V powder (5 wt.%).

If we compare the parameters and the volume of the unit cell of the  $\text{Ti}_3\text{SiC}_2$  phase and the TiC phase given in the Table 1 before and after doping with vanadium, it can be seen a significant difference: in the case of the  $\text{Ti}_3\text{SiC}_2$  phase, the volume changed from the value  $V = 142.708 \text{ \AA}^3$  to the value  $V = 141.615 \text{ \AA}^3$ , and in the case of the TiC phase, respectively, from the value  $V = 81.368 \text{ \AA}^3$  to the value  $V = 81.014 \text{ \AA}^3$ , i.e. in both cases the volume of the phases decreased significantly. This decrease in the lattice volume of both the MAX phase  $\text{Ti}_3\text{SiC}_2$  and the secondary phase TiC could be a consequence of the inclusion of vanadium atoms in the crystal lattices of the above phases. Considering that the vanadium atomic radius  $r_0 = 1.491 \text{ \AA}$  is smaller than the atomic radius of titanium  $r_0 = 1.614 \text{ \AA}$ , and it has a greater number of valence electrons than titanium, which, as is known, can contribute to an increase in the interaction of atoms of both the  $\text{Ti}_3\text{SiC}_2$  compound and the TiC compound, and as a result, a decrease in the volume of the unit cells of the aforementioned compounds.

Scanning electron microscopy was used to study the morphology of the powders of the obtained samples. The microstructures of the mixture of Ti, Si and C powders, Ti, Si and C powders with the addition of 5 wt. % Zr or 5 wt. % V after mechanochemical synthesis are presented in Fig. 4 (a-c), respectively. From Fig. 4 (a) it is clearly seen that the sample is a mixture of various particles with their size from  $0.1 \text{ \mu m}$  to  $3.8 \text{ \mu m}$  and agglomerates with sizes from  $5$  to  $10 \text{ \mu m}$ .



**Fig. 4.** Surface morphology of powder samples (a – synthesized from a mixture of Ti, Si and C powders; b – synthesized from a mixture of Ti, Si, and C powders with the addition of 5 wt. % Zr; c – synthesized from a mixture of Ti, Si, and C powders with the addition of 5 wt. % V).

From the analysis of the figures above, it can be concluded that the addition of zirconium to the mixture of Ti, Si and C does not lead to a discernable change in the size of the powder particles and its morphology: the agglomerates formed had a size from 8 to 15  $\mu\text{m}$ , and the particles – from 0.2 to 1.5  $\mu\text{m}$ . However, there were much more small particles than agglomerates, and they covered large agglomerates with a larger area.

The morphology of the powder of the mixture of Ti, Si and C with the addition of vanadium (Fig. 4, c) is noticeably different from the morphology of the powders shown in Fig. 4 (a) and 4 (b). In the case of vanadium doping, a smaller number of agglomerates with a size of 7.8 - 10  $\mu\text{m}$  is observed and a larger number of particles with a size of 2.4 to 6  $\mu\text{m}$  are present with a prevalence of particles with a size of 0.2 - 0.4  $\mu\text{m}$ .

## Conclusions

The MAX phase  $\text{Ti}_3\text{SiC}_2$  was synthesized by the mechanochemical method from the initial mixture of Ti, Si and C powders or a mixture of Ti, Si and C powders with the addition of 5 wt. % Zr or 5 wt. % V, and the influence of doping elements Zr and V on the degree of

formation of the MAX phase was investigated. It was found that the introduction of an additive of 5 wt. % V to the MAX phase  $\text{Ti}_3\text{SiC}_2$  (in contrast to the addition of 5 wt. % Zr) leads to a significant increase in the degree of its formation. That is, in the process of mechanically activated self-propagating synthesis, due to the presence of vanadium in the initial mixture of powders, the amount of the main phase  $\text{Ti}_3\text{SiC}_2$  increases and the quantity of the secondary phase TiC decreases. This could be owing to the fact that vanadium (unlike Zr) due to its catalytic and electronic properties contributed to a significant increase in the reaction rate of the formation of the  $\text{Ti}_3\text{SiC}_2$  phase, and not the TiC phase.

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## Дослідження впливу легування Zr та V MAX-фази $Ti_3SiC_2$ на ступінь її утворення методом механохімії

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В результаті механічно активованого синтезу, що самопоширюється, із вихідної суміші порошків Ti, Si, та C або суміші порошків Ti, Si, та C з додаванням 5 мас. % Zr чи з добавкою 5 мас. % V отримано MAX-фазу  $Ti_3SiC_2$ . Було досліджено вплив легування цирконієм та ванадієм MAX-фази  $Ti_3SiC_2$  на ступінь її утворення. Встановлено, що легування MAX-фази  $Ti_3SiC_2$  п'ятьма мас. % V призвело до збільшення її кількості і, відповідно, зменшення кількості вторинної фази TiC у кінцевому продукті. При легуванні MAX-фази 5 мас. % Zr навпаки її кількість зменшувалась, а кількість фази TiC збільшувалась. Методами XRD, SEM проведено якісний та кількісний рентгенофазовий аналіз, визначено параметри елементарних комірок фаз  $Ti_3SiC_2$  і TiC, вивчено мікроструктуру синтезованої MAX-фази.

**Ключові слова:** MAX-фази,  $Ti_3SiC_2$ , механохімічний метод, елементарна комірка.