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## Mechanical Alloying of Equimolar TiC-VN and TiN-VN Blends

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The investigation of step-by-step sampled products of mechanical alloying (MA) of two equimolar mixtures of TiC-VN and TiN-VN sintered with highenergy ball mill is conducted implicating number of methods of x-ray diffractometry (x-ray phase analysis, x-ray structural analysis, investigation of crystallite sizes and microstresses in the crystal lattice, etc.). As shown, the crystal structures of VN, TiN, and TiC undergo significant structural transformations during MA treatment. Specifically, part of the vanadium or titanium atoms migrate from their localization to tetrahedral pores under the impact load and part of those atoms leave their positions in the lattice and form separate clusters in the reaction zone of the milled powder. These processes lead to substantial accumulation of crystallographic point defects in phase structures presented by vacancies in metal sublattices of VN, TiN, TiC and substitutional atoms of vanadium or titanium in the crystal lattice. Overall, the increase of the portion of mentioned point defects is accompanied by the increase of microstresses in phases. Substantial portion of accumulated vacancies and some fraction of the vanadium- and titanium-atoms' clusters formed in the reaction zone of the milled powder create preconditions for further formation of mutual solid substitutional alloys. As shown, the impact of the MA treatment is decreased in the  $VN \rightarrow TiN \rightarrow TiC$  series that can be explained with such phase characteristics as dissociation energy, elasticity modulus, and thermal conductivity. The MA-activated dispersed (crystallite size up to 20 nm) powder mixture of TiC-VN acquired in the work

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may be recommended as alloying additive for medical titanium composites to increase the functional characteristics.

Key words: mechanical alloying, carbide, nitride, crystal structure, x-ray diffraction.

З використанням комплексу методів Рентґенової дифрактометрії (Рентґенової фазової аналізи, рентґеноструктурної аналізи, визначення розмірів кристалітів і мікронапружень у кристалічній ґратниці тощо) проведено дослідження покроково відібраних продуктів механохемічної (МХ) синтези у високоенергетичному планетарному млині двох еквімолярних сумішей TiC-VN і TiN-VN. В результаті показано, що в ході МХоброблення кристалічні структури VN-, ТіN- і ТіС-фаз зазнають істотних структурних перетворень, а саме, під дією ударного навантаження в структурах фаз частина атомів Ванадію чи то Титану переміщується зі своїх положень у тетраедричні пори, а частина зазначених атомів залишає відповідні ґратниці з формуванням окремих кластерів у реакційній зоні млина. Ці процеси ведуть до істотного накопичення в структурах фаз точкових дефектів у вигляді вакансій у металевих підґратницях VN, ТіN, ТіС й у вигляді занурених у кристалічні ґратниці атомів Титану чи то Ванадію. Збільшення долі зазначених точкових дефектів супроводжується збільшенням у фазах мікронапружень. В цілому наявність великої долі накопичених вакансій і певної долі кластерів атомів Ванадію та Титану, сформованих у реакційній зоні млина, створює передумови для подальшого утворення взаємних твердих розчинів заміщення. Показано, що ступінь впливу MX-оброблення зменшується в ряду  $VN \rightarrow TiN \rightarrow TiC$ , що цілком пояснюється такими характеристиками цих фаз як величина енергії дисоціяції, модуль пружности та величина термічної провідности. Одержана в роботі МХ-активована дрібнодисперсна (розмір кристалітів до 20 нм) суміш TiC-VN може бути рекомендованою в якості леґувальної добавки для поліпшення функціональних характеристик стопів медичного титану.

Ключові слова: механохемічна синтеза, карбід, нітрид, кристалічна структура, Рентґенова дифракція.

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# **1. INTRODUCTION**

Transition metal compounds such as VN and TiN nitrides as well as TiC carbide belong to the class of hard refractory metal materials according to their promising physical and chemical properties. Their high melting points, ultra-high hardness, outstanding electrical and thermal conductivity, optical properties and superior chemical stability made these compounds implemented in many applications, especially in electrochemical devices, environmental remediation, gas sensing, photocatalysis, applied ceramics, and medicine [1-5]. In particular, TiN, VN

nitrides and TiC carbide have found their successful application as individual additives to improve the functional properties of ceramics [6–10], as well as titanium alloys used for medical applications [11, 12].

*Pc*BN ceramics (60% vol. of *c*BN nitride) with binders containing well-known TiC and TiN phases combined with VN nitride [13, 14] revealed the superior performance in mechanical properties. The properties of these materials was shown to be improved to an extent, it was credited to the existence of solid solutions formed in the TiC–VN and TiN–VN binders during the HPHT (high pressure, high temperature) sintering process (7.7 GPa, 1900–2350°C). More specifically, under certain HPHT sintering modes (Ti, V)(C, N) and (V, Ti)(N, C) mutual solid solutions form in the *c*BN–TiC–VN–Al composite (ratio in % vol. as 60:17.5:17.5:5) [15] as well as (Ti, V)N and (V, Ti)N mutual solid solutions form in the *c*BN–TiC–VN–Al composite (60:17.5:17.5:5) [16].

Mechanical alloying (MA) is one of the effective methods of synthesis, which can additionally provide the formation of solid solutions, moreover, in the nanocrystalline state. The study of the TiN and VN nitrides interaction, which occur at mechanical alloying of the equimolar TiN–VN mixture in a planetary mill, reveals the formation of the (Ti, V)N and (V, Ti)N mutual solid solutions, as it occurs during HPHT sintering of this mixture [16]. The mutual solid solutions forming under mechanical alloying of TiC–ZrC mixtures were studied in [17, 18]

The purpose of this work is detailed study of the kinetics of interaction of VN nitride with TiN nitride or with TiC carbide during mechanical alloying of the equimolar TiC–VN and TiN–VN mixtures in a highenergy planetary mill.

## 2. EXPERIMENTAL/THEORETICAL DETAILS

Two equimolar TiC-VN and TiN-VN mixtures of starting powders containing VN and TiN or TiC were placed into two steel vials for further ball milling in a high-energy planetary mill. Steel balls (diameter of 10 mm) were used for the milling with the balls to powder mass ratio of 20:1. The regime was chosen as 10 min of treatment and 20 min of cooling. The temperature of the working area in the reaction zone did not exceed 100°C during experiment; the ball-mill rotation speed was 1400 rpm.

Phase transformations in TiC–VN and TiN–VN mixtures processed have been studied by x-ray diffraction methods on the test samples selected after every 60 min of milling. XRD data was collected with  $\square$ POH-3M automatic diffractometers (Cu $K_{\alpha}$  radiation) in a discrete mode under the following scanning parameters: observation range  $2\theta = 20^{\circ}-100^{\circ}$ , step scan of 0.05° and counting time per step at 3 s. The original software package [15], including full complex of standard Rietveld procedures, has been used for analysis and interpretation of the x-ray diffraction patterns obtained, namely, determination of both peak positions and integral intensities of the Bragg reflections by means of full profile analysis; carrying out qualitative and quantitative phase analysis using the least square method for lattice parameters refinement; testing of the structure models proposed and refining crystal structure parameters (including coordinates of atoms, atomic position filling, texture, *etc.*); calculation of the parameters of the real structure of the individual phases (coherent block sizes and microdeformation values).

#### **3. RESULTS AND DISCUSSION**

The results of the XRD phase analysis reveal that the test samples of both TiC–VN and TiN–VN mixes contain initial VN and TiC or TiN phases with a small amount of steel ball material contamination in the final products (Fig. 1). However, the lattice parameters of these phases differ from those in the initial blends (Table 1). The characterization of changes in lattice parameters during MA treatment (a(t) values) was controlled by the relative value of their distortions, which were calculated according to the formula:  $\varepsilon[\%] = 100(a_t - a_0)/a_0$ , where  $a_t$  is the value of the lattice parameter at a certain t milling time, and  $a_0$  is in the initial mixture (Table 1, Fig. 2). It can be seen that the crystal lattice distortions of TiN and VN nitrides are much greater than those ones of TiC carbide.

To clarify the causes of changes in  $\epsilon$  values (Fig. 2), first the refinement of the crystal structures of TiC, TiN and VN phases within the



Fig. 1. Diffraction patterns of the TiC–VN and TiN–VN equimolar blends after 5 hours of treatment in a planetary mill.

**TABLE 1.** Parameters, distortions and microdeformations of the lattices of the TiC, TiN and TiC phases. It is impossible to determine the value of microdeformation of the lattices of nitrides existing in the TiN–VN blend due to the reflections overlap of these phases (Fig. 1).

Mill- ing time t, h	Lattice pa- rameter a, nm	Lattice distortion ε, %	$\begin{array}{c} \text{Micro-} \\ \text{defor-} \\ \text{mation} \\ \Delta d/d, \% \end{array}$	Lattice pa- rameter a, nm	Lattice distortion ε, %	Micro- defor- mation $\Delta d/d$ , %	
			TiC-VN bl	end			
	TiC carbide			VN nitride			
0	0.43252(3)	0	0	0.41251(6)	0	0	
1	0.43246(3)	-0.0139	0.13(1)	0.41197(6)	-0.1302	0.20(4)	
2	0.43242(4)	-0.0231	0.15(2)	0.41142(7)	-0.2642	0.40(5)	
3	0.43240(6)	-0.0277	0.14(2)	0.41089(9)	-0.3927	0.46(8)	
4	0.43266(9)	0.0324	0.12(1)	0.4112(1)	-0.3176	0.39(4)	
<b>5</b>	0.43271(9)	0.0439	0.00(2)	0.4116(1)	-0.2254	0.25(2)	
TiN-VN blend							
	ŋ	l'iN nitride			VN nitride		
0	0.42440(5)	0	_	0.41251(6)	0	_	
1	0.42401(8)	-0.0919	_	0.41230(9)	-0.0509	_	
2	0.42293(9)	-0.3464	-	0.41207(9)	-0.1067	_	
3	0.4221(1)	-0.5419	-	0.4117(1)	-0.2061	_	
4	0.4218(1)	-0.6126	-	0.4120(1)	-0.1139	_	
<b>5</b>	0.4215(1)	-0.6833	_	0.4135(1)	0.2400	_	

NaCl type structure model (space group Fm3m (No. 225)) has been conducted: 4 Ti or 4 V in 4*a* 0 0 0; 4 N or 4 C atoms in 4*b* 0.5 0.5 0.5. As a result of these calculations, it was shown that the 4*a* position, which is occupied by metal atoms, becomes vacant at almost complete filling of the 4*b* position with nitrogen or carbon atoms. However, the reliability factor  $R_B$  exceeded the value of 0.03.

To improve the value of the  $R_{\rm B}$  factor, several structural models have been proposed and tested for the TiC, TiN, VN phases existing in MA treated TiC–VN and TiN–VN mixtures. The best correlation between the calculated and experimental intensities values of the diffraction pattern reflections were obtained for the models presented in Table 2 ( $R_B < 0.01$ ).

The obtained results (Table 2) indicate that the crystal structure of the VN nitride undergoes certain transformations during the MA processing. More specifically, some vanadium atoms leave the metal sub-



**Fig. 2.** Distortion of the crystal lattices of TiC, TiN and VN phases during MA treatments of TiC–VN blend (*a*) and TiN–VN blend (*b*) in a planetary mill.

lattice, which accommodates the V(1) atoms and the available Vac V(1) vacancies (position 4a). One part of the displaced atoms is located in the tetrahedral pores of the VN nitride lattice (V(2) atoms in the 4e position), and the second part of these atoms leaves the VN nitride with the formation of separate vanadium clusters in the reaction zone. Figure 3, a illustrates the transformation of the crystal structure of the VN nitride during the MA treatment. As it can be seen on Fig. 3, a, the main vanadium atoms in the VN nitride are surrounded by 6 nitrogen atoms at a distance of 0.206 nm (1 octahedron), and the distance of a possible migration of a vanadium atom into a tetrahedral pore (3 tetrahedron) is 0.180 nm (the arrow in Fig. 3, a), respectively.

Similar migrations caused by MA treatment are also characteristic of titanium atoms in TiN nitride and TiC carbide (Fig. 3, *b*). However, unlike to the cubic structure of VN, the best result was obtained for the model of the rhombohedral structure (presentation of the structures of TiN and TiC along their body diagonal, Table 2), in which the tetrahedral pore (tetrahedron 3-Ti(2)Ti(1)<sub>4</sub>) becomes deformed, and the migration distances of titanium atoms vary from  $\approx 0.18$  nm (three positions) to  $\approx 0.24$  nm (one position).

Obviously, the difference in the possible positions, intended for placement of the vanadium atoms (regular tetrahedron  $3-V(2)V(1)_4$ ) or titanium atoms (a triangle  $3-Ti(2)Ti(1)_3$ , in fact), results from the peculiarities of directionality atomic bonds in vanadium or titanium.

According to the results of crystal-structure refinement, which was carried out within the framework of the proposed models (Table 2), it was determined the quantitative content of titanium or vanadium both in the metal sublattice of the structures (positions 3a and 4a) and in the tetrahedral pores where these atoms migrated under the shock

load. The data obtained in this manner for TiC and VN phases that exist in the MA treated TiC-VN blend are presented in Fig. 4. It should be noted that analogical plots for the phases that exist in the MA treated TiN-VN blend are of the same kind. The total metal content in each of the VN, TiN, and TiC phases is presented in Fig. 5.

A detailed XRD study of the crystal structures of the VN, TiN, TiC phases, which exist in the step-by-step milled products, revealed certain tendencies in the kinetics of mechanochemical synthesis of equimolar TiC-VN and TiN-VN mixtures under MA processing in a planetary mill, as well as in the nature of the interaction of the phases existing in these blends.

Moreover, accumulation of defects in the metal sublattices of TiC, TiN, VN phases occurs mainly in the first stage of MA processing (up

TiC carbide in TiC–VN blend							
Atom	Position	Occupation	X	Y	Z		
Ti(1)	3a	0.778(1)	0	0	0		
Ti(2)	3a	0.222(1)	0	0	0.368(1)		
С	3a	1.000(1)	0	0	0.5		
Vac in Ti(1)	3a	-0.028(2)	0	0	0		
	Space grou	up		R3m (No. 160)			
Lat	tice parameter	rs a, c nm		0.3057(2), 0.74	482(2)		
Tota	l isotropic <i>B</i> f	actor, nm²		2.56(3).10	-2		
Cal	lculated conte	nt, at.%		$49.3\mathrm{Ti}+50.7\mathrm{N}$			
Reliability factor				$R_B = 0.008$			
		VN nitride i	n TiC–VN blend	l			
Atom	Position	Occupation	Х	Y	Z		
V(1)	4a	0.980(2)	0	0	0		
V(2)	4e	0.020(2)	0.25	0.25	0.25		
Ν	4b	1.000(1)	0.5	0.5	0.5		
Vac in V(1)	4a	-0.126(4)	0	0	0		
Space group				<i>F</i> -43 <i>m</i> (No. 216)			
La	attice paramet	er a nm		0.4116(1)			
Tota	l isotropic <i>B</i> f	actor, nm²		$3.69(1) \cdot 10^{-2}$			
Cal	lculated conte	nt, at.%		$47.0\mathrm{V}+53.0\mathrm{N}$			
	Reliability fa	actor		$R_B = 0.004$			

**TABLE 2.** Crystal structure data for VN and TiC or TiN phases existing in TiC–VN and TiN–VN blends treated for 5 h in planetary mill.

TiN nitride in TiN–VN blend						
Atom	Position	Occupation	Х	Y	Z	
Ti(1)	3a	0.783(1)	0	0	0	
Ti(2)	3a	0.217(1)	0	0	0.356(1)	
Ν	3a	1.000(1)	0	0	0.5	
Vac in Ti(1)	3a	-0.034(2)	0	0	0	
	Space grou	up		<i>R</i> 3 <i>m</i> (No. 160)		
Lat	tice paramete	rs <i>a</i> , <i>c</i> nm		0.2983(1), 0.73	313(2)	
Total isotropic $B$ factor, $\mathrm{nm}^2$				2.25(3).10	-2	
Calculated content, at. $\%$				49.1Ti+50.9N		
Reliability factor				$R_B = 0.008$		
		VN nitride ir	n TiN–VN bler	ıd		
Atom	Position	Occupation	Х	Y	Z	
V(1)	4a	0.955(2)	0	0	0	
V(2)	4e	0.045(2)	0.25	0.25	0.25	
Ν	4b	1.000(1)	0.5	0.5	0.5	
Vac in V(1)	4a	-0.227(4)	0	0	0	
Space group				<i>F</i> -43 <i>m</i> (No. 216)		
Lattice parameter a, nm				0.4135(2)		
Total isotropic $B$ factor, nm <sup>2</sup>				$2.55(1) \cdot 10^{-2}$		
Calculated content, at. $\%$				$\mathbf{43.6~V} + \mathbf{56.4~N}$		
Reliability factor				$R_B = 0.009$		

Contin	nuation	Τ	'able	2.

to 3 hours), as can be seen in Fig. 4. In this case, one part of the metal atoms leaves the crystal lattice forming the atomic clusters in the reaction zone of the milled powder, while the other part of the atoms migrates into tetrahedral pores of the crystal structure. At the first stage of milling, there is also a gradual increase in the microdeformation of the crystal lattice, which correlates with an increase in its distortions (Table 2, Fig. 5) as well as with an increase in the dislocation density (it is impossible to determine the value of microdeformation of the lattices of nitrides existing in the TiN–VN blend due to the reflections overlap of these phases (Fig. 1)).

After 3 hours of milling of TiC–VN and TiN–VN mixtures in a planetary mill, the process of active migration of vanadium atoms into tetrahedral pores of VN stops, at the same time the process of migration of titanium atoms into tetrahedral pores of TiC and TiN slows down



Fig. 3. Arrangement of the atoms in the structures of the phases that exist in TiC-VN and TiN-VN mixtures treated in a planetary mill for 5 hours: VN nitride (a), TiN nitride or TiC carbide (b). Atoms marked as follow: metal atoms are large black circles, nitrogen or carbon atoms are medium-coloured circles, and metal atoms jumped from the main position are small light grey circles. Polyhedra:  $1-Me(1)N_6$  or  $1-Ti(1)C_6$ ,  $2-Me(1)Me(2)_4$ ,  $3-Me(2)Me(1)_4$ .

(Fig. 4). This causes a decrease in the deformation of the crystal lattices of TiN, VN nitrides and TiC carbide (Fig. 6), although their lattice parameters continue to change (Table 1, Fig. 2). In our opinion, this may be caused by the beginning of processes wherein the available vacancies in the metal sublattices of these phases are filled with free atoms of titanium or vanadium that have entered the reaction zone of the milled powder. Unfortunately, due to the closeness of the atomic scattering functions of titanium and vanadium, it is impossible to determine correctly the amount of dissolved metal by XRD method. But the formation of mutual solid solutions is evidenced by an increase in the parameters of the VN lattices (the size of titanium atom is large than vanadium one), as well as the decrease in the parameters of the TiC and TiN lattices which goes on despite the decrease in the amount of vacancies (Table 1, Fig. 4).

As it is known, the basis of mechanochemical synthesis is the mechanical processing of solid mixtures, during which grinding and plastic deformation of substances occur. Under the effect of applied mechanical stresses, the process of formation of point defects is initiated in the material, which leads to partial destruction of the crystal structure accompanied by creation of active centres for subsequent chemical



**Fig. 4.** Plots of dependences of the amount of available vacancies in the metal sublattice of the TiC and VN phases existing in the MA treated TiC–VN mixture (circles), as well as of the amount of atoms that have moved into the tetrahedral pores (triangles).

reactions. These are the processes, which occur during the MA processing of TiC–VN and TiN–VN blends in a high-energy planetary mill. More specifically, they are as follow.

1. At the first stage of MA synthesis (up to 3 hours of processing), there is an active accumulation of point defects, namely, an increase in vacancies in the metal sublattice of crystal structure as well as an increase in the number of atoms in its internodes (Fig. 4).



Fig. 5. Dependence of the content of metals in the crystal structures of TiC, TiN, VN phases on the MX processing time of TiC–VN and TiN–VN mixtures in a planetary mill.

Phase	Melting point $T$ , °C	Dissociation energy $D_0,{ m kJ/mole}$	Young's modulus, GPa	Thermal conductivity, W/(m·K)
TiC	3260	1388	494	7
TiN	2930	1261	251	29
VN	2050	1197	210	60-120

**TABLE 3.** Some characteristics of the phases included in the TiC–VN and TiN–VN charges.

At the same time, clusters of vanadium and titanium atoms appear in the reaction zone of the milled powder, which have left the structures of the TiN and VN phases, as it evidenced by a shift in their composition (Fig. 5). Moreover, the process of forming titanium clusters during MA treatment of the TiC-VN mixture is slower. The accumulation of defects is accompanied by an increase in microstresses of crystal lattices and by an increase in the dislocation density (Fig. 6).

2. At the second stage of MA synthesis (after 3 hours of processing), the stress field created at the first stage as a result of the accumulation of point defects is starting to gradually relax (Fig. 6). It is probably due to a decrease in the number of atoms placing in the internodes (Fig. 4). Obviously, the interatomic bonds of these atoms in the structures are somewhat weaker, which contributes to the mass transfer to the reaction zone of the milled grains. The set of available factors (a high proportion of vacancies in the metal sublattice of the phases, as well as some proportion of the atomic clusters formed, Fig. 4) facilitate the creation of active centres for the further formation of mutual solid solutions. It is evidently illustrated by exceeding the lattice value of the VN nitride over its value in the initial charge (Fig. 2, b).

3. The extent of exposure of mechanochemical processing on the VN or TiN nitrides and on the TiC carbide, used in these experiments, is fully explained by the characteristics of these phases given in Table 3. Namely, the VN nitride was shown to be the most susceptible to MA treatment (especially in the presence of TiN but not of TiC). It can be explained by the increase in the values of the dissociation energy, melting temperature, and Young's modulus as  $VN \rightarrow TiN \rightarrow TiC$ . It should also be noted that the decrease in thermal conductivity as  $VN \rightarrow TiN \rightarrow TiC$  also influences on the extent of exposure of the MA synthesis process of TiC–VN and TiN–VN blends.

# 4. CONCLUSIONS

Using a wide complex of XRD methods, the products of mechanochem-



Fig. 6. Dependences of distortions and microdeformations of the crystal structures of TiC (a) and VN (b) phases as well as dislocation density (c) on the milling time of the TiC–VN mixture in a planetary mill.

ical processing of equimolar TiC–VN and TiN–VN mixtures were studied systematically. Studies of the phase transformation kinetics were performed and revealed the following.

1. The crystal structures of VN, TiN, TiC phases undergo certain transformations during the MA processing. Under the impact load, some vanadium or titanium atoms move into the tetrahedral pores of the corresponding lattices, and some of these atoms leave the lattices of nitrides or carbide forming the separate clusters in the reaction zone of the powder.

2. Accumulation of the indicated point defects in the crystal lattices of VN, TiN, TiC phases occurs abundantly at the first stage of MA processing (up to 3 hours of treatment). This process is accompanied by a gradual increase in microstresses values and an increase in the dislocation density.

3. At the second stage of the MA process (after 3 hours of treatment),

the stress field created at the first stage as a result of point-defects' accumulation is gradually starting to relax. The presence of a large proportion (share) of vacancies in the structures and a certain proportion (share) of formed atomic clusters in the reaction zone of the powder contribute to the creation of active centres for further formation of mutual solid solutions on their basis.

4. The extent of exposure of mechanochemical processing on the VN, TiN, TiC phases decreases in the series  $VN \rightarrow TiN \rightarrow TiC$ , which is fully explained by such characteristics of these phases, as values of the dissociation energy, the Young's modulus, the thermal conductivity.

5. Fine-crystalline (crystallite size up to 30 nm) MA activated powders of an equimolar TiC–VN mixture will be tested as functional additives to improve the properties of medical titanium alloys.

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