UDC 666.3:621.762
IRSTI 53.37.31, 61.35.31.

https://doi.org/10.55452/1998-6688-2026-23-3-434-447

[1*]Zhumazhanova A.T.,
PhD, Junior Researcher, ORCID ID: 0000-0002-5483-9552,
*e-mail: ainashzhumazhanova@gmail.com
[1]Shakirzyanov R.I.,
Cand. Phys.-Math. Sci., Junior Researcher, ORCID ID: 0000-0001-9908-3034,
e-mail: shakirzyanov_ri@enu.kz

[1]L.N. Gumilyov Eurasian National University, Astana, Kazakhstan

EFFECT OF TANTALUM AND VANADIUM CARBIDE DOPANTS ON THE STRUCTURE AND MECHANICAL PROPERTIES OF ALUMINUM NITRIDE-BASED CERAMICS

Abstract

This work investigates the effect of tantalum carbide and vanadium carbide additions on the phase composition, microstructure, and mechanical properties of AlN-based ceramic composites synthesized by mechanochemical solid-state processing followed by thermal treatment. The molar fraction of the carbide additives was varied within the range of x = 0.01-0.10. Scanning electron microscopy combined with energy-dispersive X-ray spectroscopy revealed a homogeneous distribution of the reinforcing phases after mechanochemical activation. X-ray diffraction analysis showed that TaC remained chemically stable during processing, leading to the formation of a two-phase AlN-TaC composite with reduced lattice distortions of the AlN matrix. In contrast, VC underwent partial oxidation during heat treatment, resulting in the formation of secondary V_2O_5 and a different phase evolution. Mechanochemical processing produced a more homogeneous powder morphology and suppressed the formation of large carbide agglomerates, providing favorable conditions for subsequent microstructural development. Mechanical testing demonstrated that a pronounced improvement in hardness and flexural strength occurred at dopant contents above x = 0.03, with the greatest strengthening effect observed for the TaC-containing composites owing to the retention of a stable, uniformly dispersed high-hardness carbide phase. The enhanced mechanical performance is attributed to the combined effects of dispersion strengthening, inhibition of grain growth, grain-boundary strengthening, and crack-deflection mechanisms. These results establish clear relationships between processing, phase evolution, microstructure, and mechanical behavior, demonstrating the potential of mechanochemical processing for the development of high-performance AlN-based ceramic composites intended for demanding structural and high-temperature applications.

Keywords: aluminum nitride, carbonitride ceramics, mechanochemical synthesis, tantalum carbide, vanadium carbide, phase composition, mechanical properties.

Received April 30, 2026; revised August 3, 2026; accepted August 10, 2026.

Introduction

Aluminum nitride (AlN) is recognized as one of the most promising structural and functional ceramic materials owing to its unique combination of high thermal conductivity, excellent electrical insulation, low dielectric constant, high-temperature stability, and chemical inertness [1–4]. These characteristics make AlN highly attractive for applications in power electronics, thermal management systems, electronic packaging, aerospace engineering, and other advanced technologies operating under severe thermal and mechanical conditions. In addition to its excellent thermal performance, AlN possesses a relatively low coefficient of thermal expansion that closely matches that of silicon, making it an important substrate material for modern semiconductor devices [1–4]. Consequently, the development of AlN-based ceramics with improved structural reliability while preserving their

functional properties remains an important research direction in advanced ceramic materials. The development of such materials is driven by the increasing demand for ceramic components capable of operating under high temperatures, severe mechanical loading, and aggressive environments, including thermal barrier systems, wear-resistant components, protective structural materials, and electronic devices requiring high thermal stability [5].

Despite these advantages, the broader application of monolithic AlN as a structural ceramic is still restricted by its relatively low fracture resistance and moderate mechanical strength. It is well established that the performance of AlN ceramics depends not only on the intrinsic properties of the AlN crystal lattice but also on microstructural factors, including grain size, grain boundary chemistry, residual porosity, oxygen-related defects, and the presence of secondary phases. Accordingly, controlling the microstructure through compositional modification and optimization of processing routes has become one of the most effective strategies for simultaneously improving mechanical performance while maintaining the functional characteristics of AlN ceramics.

Among the various approaches proposed to enhance the mechanical properties of AlN, the incorporation of dispersed reinforcing particles has attracted considerable attention. Previous studies have demonstrated that secondary phases can effectively inhibit grain growth during sintering, promote microstructural refinement, modify grain boundary characteristics, and improve resistance to crack initiation and propagation through dispersion strengthening mechanisms [6–10]. Recent investigations have further confirmed that optimization of the reinforcing phase and processing conditions significantly affects densification behavior, grain morphology, and the overall mechanical performance of AlN-based ceramics [11–12]. These findings indicate that both the nature of the reinforcing phase and the processing route play decisive roles in determining the final properties of ceramic composites.

Among refractory ceramic compounds, transition metal carbides are particularly attractive because of their exceptional hardness, high elastic modulus, excellent thermal stability, and wear resistance. Tantalum carbide (TaC), belonging to the family of ultra-high-temperature ceramics, possesses an extremely high melting point (~3880 °C), high hardness, and excellent thermodynamic and oxidation stability, making it a promising reinforcing phase for ceramic matrices [13–15]. Vanadium carbide (VC) also exhibits high hardness and good mechanical characteristics; however, compared with TaC, it has lower oxidation resistance and higher chemical reactivity at elevated temperatures [16, 17]. These differences suggest that TaC and VC may affect phase evolution, microstructure development, and strengthening mechanisms of AlN ceramics in different ways. Although other refractory carbides, including TiC, ZrC, HfC, and NbC, have also been investigated as reinforcing phases for ceramic composites, TaC and VC were selected in the present study because they represent carbides with distinctly different thermodynamic stability and oxidation behavior while maintaining similarly high hardness and refractory characteristics. This makes them suitable model additives for investigating how the intrinsic properties of refractory carbides influence phase evolution, microstructure development, and mechanical strengthening of AlN ceramics under identical processing conditions.

Another important factor governing the structure and properties of ceramic composites is the powder processing route. Mechanochemical activation has proven to be an effective approach for improving powder homogeneity, increasing the contact area between constituent particles, enhancing powder reactivity, and promoting solid-state reactions during subsequent heat treatment [18, 19]. Compared with conventional powder mixing, mechanochemical processing facilitates a more uniform distribution of reinforcing particles and contributes to the formation of finer and more homogeneous microstructures. These features are particularly important for ceramic composites containing dispersed reinforcing phases, where the uniformity of particle distribution strongly influences phase evolution and the resulting mechanical performance.

Although previous studies have demonstrated that refractory carbide additions can improve the mechanical behavior of ceramic materials, the comparative influence of TaC and VC on the phase evolution, microstructure formation, and strengthening mechanisms of AlN ceramics prepared by

mechanochemical synthesis has not yet been systematically investigated. In particular, the relationship between carbide type, oxidation behavior during heat treatment, phase composition, and the resulting mechanical properties remains insufficiently understood. Addressing this knowledge gap is essential for the rational design of AlN-based ceramic composites with improved structural reliability [20, 21].

Therefore, the aim of the present work is to investigate the influence of TaC and VC additions on the phase composition, microstructure, and mechanical properties of AlN ceramics prepared by mechanochemical synthesis followed by heat treatment in air. Particular attention is devoted to clarifying the relationships between carbide type, phase evolution, microstructure development, and strengthening mechanisms, as well as explaining the different reinforcing efficiencies of TaC and VC under identical processing conditions.

Materials and methods

Commercial aluminum nitride (AlN) powder (Sigma-Aldrich, St. Louis, MO, USA) was used as the matrix material, whereas commercial tantalum carbide and vanadium carbide powders (Sigma-Aldrich, St. Louis, MO, USA) served as reinforcing additives. Based on previous studies on AlN-based ceramic composites, TaC and VC were introduced in molar fractions of x = 0.01, 0.03, 0.05, and 0.10 to systematically investigate the influence of carbide content on the phase composition, microstructure, and mechanical properties of the composites. This concentration range was selected to cover both low and relatively high additive contents while enabling evaluation of composition-property relationships without excessive modification of the AlN matrix.

The composite powders were prepared by a mechanochemical solid-state route using a Fritsch PULVERISETTE 6 planetary ball mill (Fritsch, Berlin, Germany). Appropriate amounts of TaC or VC were mixed with the AlN powder and mechanically activated for 30 min at a rotational speed of 250 rpm. Milling was carried out in an 80 ml tungsten carbide (WC) vial using 22–24 tungsten carbide balls with a diameter of 10 mm. The total powder mass was 20 g. These milling conditions were selected to ensure efficient homogenization of the powder mixture while avoiding excessive deformation and cold welding during mechanochemical processing.

Following mechanochemical activation, the powders were heat-treated in air using a SNOL 7.2/1100 muffle furnace (SNOL, Utena, Lithuania) at 500 °C for 5 h. This treatment was performed to stabilize the activated powders prior to structural and mechanical characterization.

The morphology and microstructure of the synthesized composites were examined using a Phenom ProX scanning electron microscope (Thermo Fisher Scientific, Eindhoven, The Netherlands) operated at an accelerating voltage of 10–15 kV under high-vacuum conditions. The elemental composition and spatial distribution of Al, N, Ta, V, C, and O were investigated by energy-dispersive X-ray spectroscopy (EDS) using the integrated detector, and elemental maps were recorded to evaluate the homogeneity of the reinforcing phases within the AlN matrix.

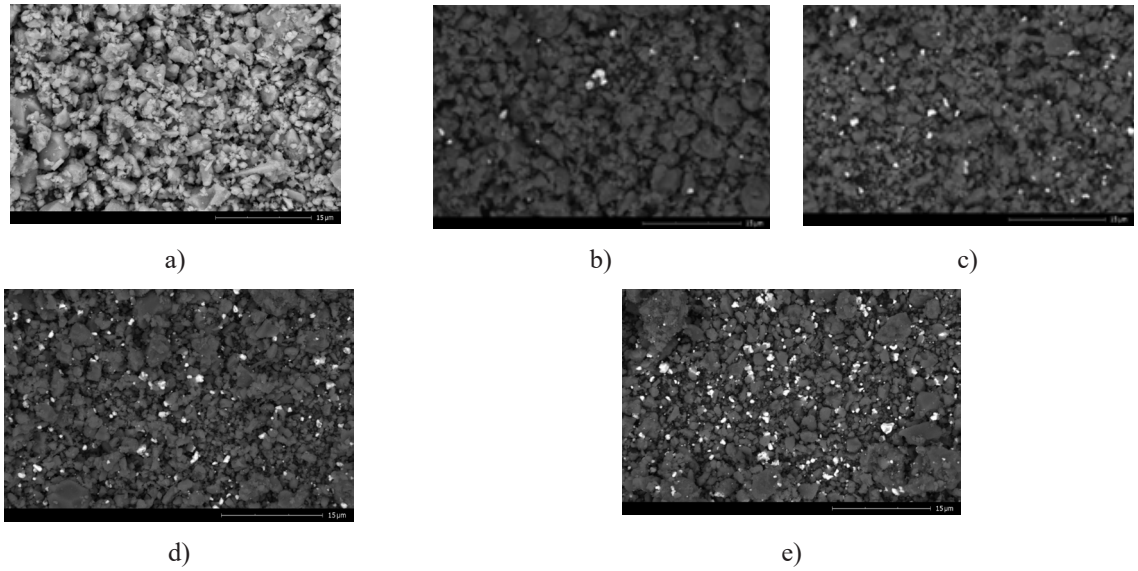
Phase composition was analyzed by X-ray diffraction (XRD) using a Bruker D8 ADVANCE ECO diffractometer (Bruker, Karlsruhe, Germany). Diffraction patterns were collected over the 2θ range of 20–80° with a step size of 0.02°. Phase identification was carried out using the DIFFRAC. EVA software package (Bruker AXS), and the lattice parameters were determined from the processed diffraction data.

The mechanical properties of the ceramic composites were evaluated by measuring the Vickers microhardness under indentation loads of 0.5–2 N. The flexural strength was determined using the three-point bending method.

Results and discussion

SEM micrographs of the initial AlN powder and the (1-x)AlN-xTaC composite powders after mechanochemical treatment are presented in Figure 1. The initial AlN powder (Figure 1a) consists

of irregularly shaped particles with a relatively broad particle-size distribution and several coarse agglomerates, which is typical of commercially available ceramic powders. Following the addition of TaC and high-energy milling (Figures 1b-e), the morphology becomes progressively more homogeneous, accompanied by a noticeable reduction in the size and number of coarse agglomerates.



a) b) c)

d) e)

a) initial; b) at addition of 0.01 M; c) at addition of 0.03 M;
d) at addition of 0.05 M; e) at addition of 0.10 M

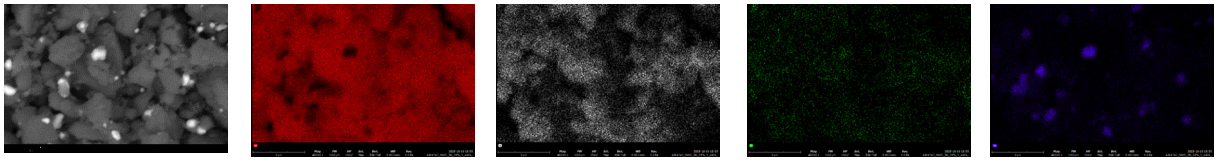
Figure 1 – SEM micrographs of the initial AlN powder and (1-x)AlN-xTaC composite powders after mechanochemical activation with different TaC. All micrographs were recorded at the same magnification

The bright particles visible in the SEM images correspond to the Ta-containing phase because of the higher atomic-number contrast relative to the AlN matrix. No pronounced carbide agglomeration is observed over the investigated composition range, indicating that the selected milling conditions ensured efficient dispersion of the reinforcing phase throughout the powder mixture.

The observed morphological evolution is consistent with the well-established mechanisms of mechanochemical processing. During high-energy milling, repeated particle fracture, cold welding, and intensive mixing increase the contact area between AlN and TaC particles, thereby improving powder homogeneity and promoting intimate interparticle contact. Such structural homogenization is expected to facilitate subsequent solid-state reactions during heat treatment and contribute to the formation of a more uniform ceramic microstructure.

Although no quantitative particle-size analysis was performed, the SEM observations indicate a reduction in particle agglomeration and a more homogeneous distribution of the reinforcing phase after mechanochemical treatment. Such a microstructural arrangement is generally considered favorable for efficient load transfer between the ceramic matrix and the reinforcing particles and may therefore contribute to the enhanced mechanical properties discussed in the following sections.

To further evaluate the distribution of the reinforcing phase, elemental mapping was performed by energy-dispersive X-ray spectroscopy (EDS). Representative elemental maps of the (1-x)AlN-xTaC composite with x = 0.10 are shown in Figure 2, while the average elemental compositions determined from three independently analyzed regions are summarized in Table 1.



a) b) c) d) e)
a) SEM image; b) Al; c) N; d) C; e) Ta

Figure 2 – SEM micrograph and corresponding EDS elemental maps of the (1-x)AlN-xTaC composite with x = 0.10

Table 1 – Average elemental composition of the (1-x)AlN-xTaC composites determined by EDS analysis (mean ± standard deviation, n = 3)

Composition	C (wt.%)	N (wt.%)	Al (wt.%)	Ta (wt.%)
AlN (x = 0)	–	40.20 ± 0.44	59.80 ± 0.44	–
(1-x)AlN-xTaC (x = 0.01)	2.53 ± 0.85	37.97 ± 2.97	56.80 ± 1.82	2.70 ± 1.27
(1-x)AlN-xTaC (x = 0.03)	2.93 ± 2.56	40.97 ± 2.08	53.93 ± 4.28	2.16 ± 0.99
(1-x)AlN-xTaC (x = 0.05)	3.37 ± 1.43	41.67 ± 1.20	50.10 ± 3.68	4.87 ± 1.12
(1-x)AlN-xTaC (x = 0.10)	5.03 ± 3.47	39.58 ± 0.93	48.00 ± 4.70	6.74 ± 1.96

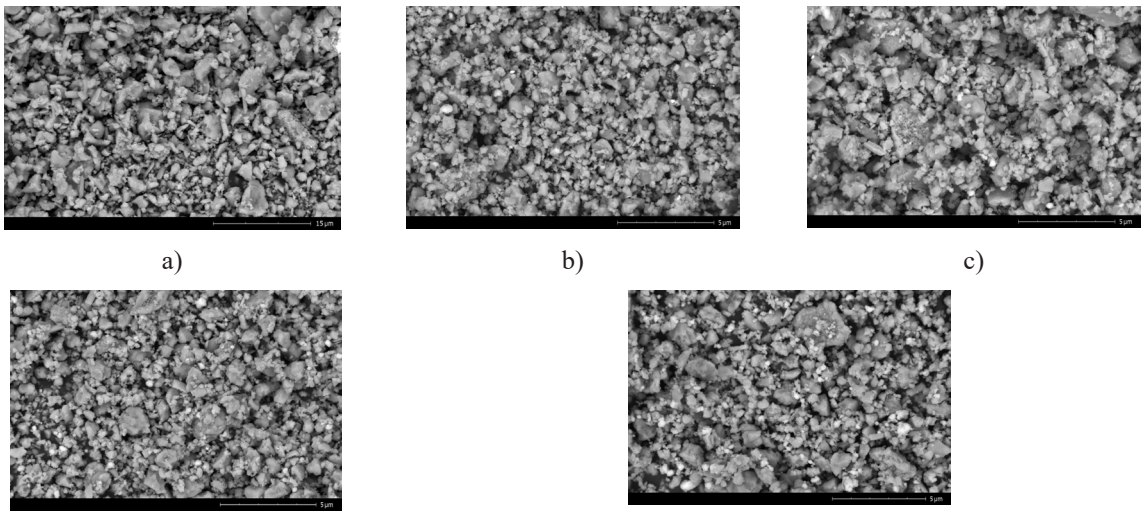
The elemental maps reveal a relatively uniform spatial distribution of Al, N, C, and Ta throughout the analyzed region without evidence of pronounced elemental segregation. The spatial coincidence of carbon and tantalum confirms that the bright particles correspond to the TaC reinforcing phase, whereas the continuous distribution of Al and N demonstrates that the addition of TaC does not disrupt the continuity of the AlN matrix.

The quantitative EDS results exhibit good reproducibility and show an overall increase in the Ta content with increasing nominal TaC concentration. Minor deviations between the nominal and measured compositions can be attributed to the local nature of EDS analysis and the heterogeneous distribution of carbide particles within the analyzed regions. Nevertheless, the relatively small standard deviations and the absence of pronounced compositional fluctuations confirm that mechanochemical processing provides efficient mixing of the starting powders and promotes a homogeneous dispersion of the reinforcing phase throughout the AlN matrix.

SEM micrographs of the initial AlN powder and the (1-x)AlN-xVC composite powders after mechanochemical treatment are presented in Figure 3. Similar to the TaC-containing composites, the initial AlN powder consists of irregularly shaped particles forming coarse agglomerates. After the addition of VC and mechanochemical activation (Figures 3b-e), the powder mixtures exhibit a relatively homogeneous particle distribution without pronounced changes in the overall particle morphology.

In contrast to the AlN-TaC system, the incorporation of VC does not produce pronounced morphological refinement of the powder mixture. The overall particle size and agglomeration state remain comparable to those of the initial AlN powder, although mechanochemical treatment provides satisfactory homogenization of the powder mixture. No large agglomerates of the reinforcing phase are observed within the investigated concentration range.

The limited influence of VC on the powder morphology suggests that, under the selected milling conditions, the principal effect of mechanochemical processing is associated with improved dispersion of the reinforcing particles rather than significant particle fragmentation. Nevertheless, repeated fracture, cold welding, and intensive mixing during high-energy milling are expected to enhance the contact between AlN and VC particles, thereby promoting structural homogeneity before subsequent heat treatment.



a) b) c)

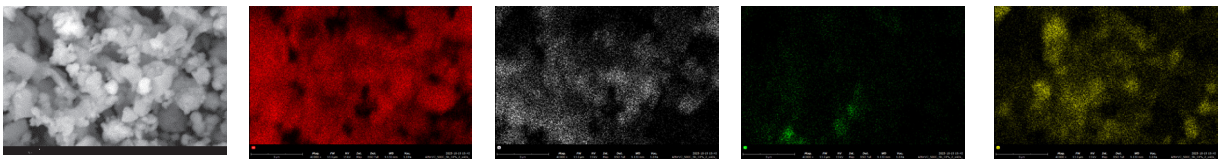
d) e)

a) initial; b) at addition of 0.01 M; c) at addition of 0.03 M;
d) at addition of 0.05 M; e) at addition of 0.10 M

Figure 3 – SEM micrographs of the initial AlN powder and (1-x)AlN-xVC composite powders after mechanochemical activation with different VC. All micrographs were acquired at the same magnification

To further evaluate the elemental distribution, energy-dispersive X-ray spectroscopy (EDS) elemental mapping was performed for the (1-x)AlN-xVC composite with x = 0.10. The corresponding SEM image and elemental maps are presented in Figure 4, while the quantitative EDS results are summarized in Table 2.

The elemental maps demonstrate a relatively uniform distribution of Al, N, C, and V throughout the analyzed region without evidence of pronounced elemental segregation. The overlap between the carbon and vanadium signals indicates that the reinforcing particles remain uniformly dispersed within the AlN matrix after mechanochemical treatment. Furthermore, the continuous distribution of Al and N confirms that the incorporation of VC does not disturb the integrity of the ceramic matrix.



a) b) c) d) e)

a) SEM image; b) Al; c) N; d) C; e) V

Figure 4 – SEM micrograph and corresponding EDS elemental maps of the (1-x)AlN-xVC composite with x = 0.10

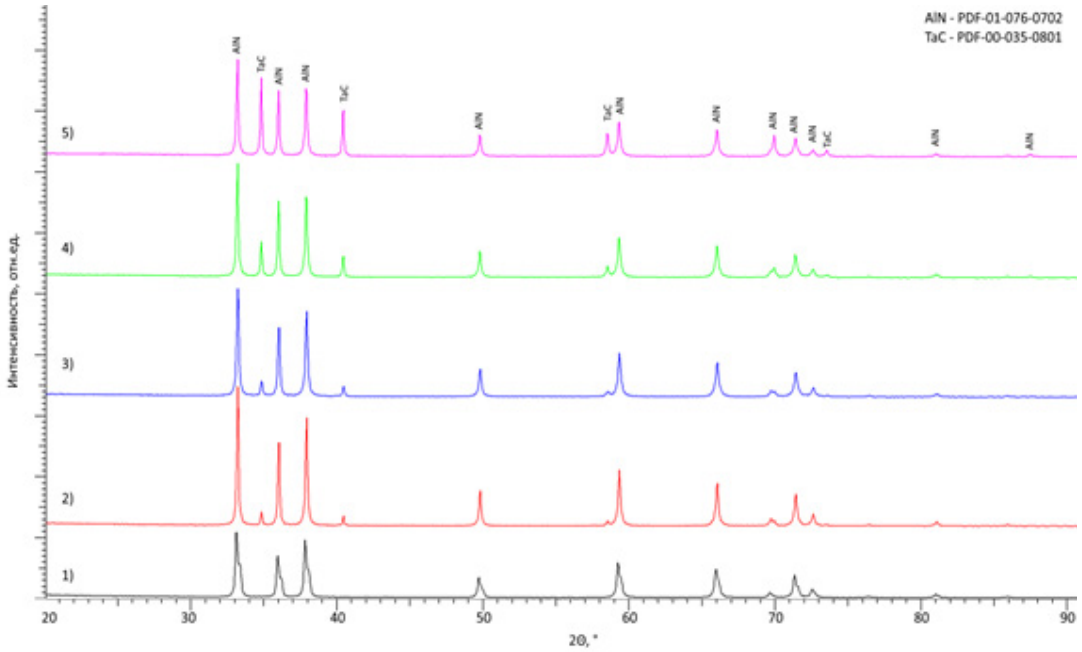
The quantitative EDS analysis exhibits good reproducibility and an overall increase in the measured V content with increasing nominal VC concentration. Minor differences between the nominal and measured compositions are attributed to the local nature of EDS analysis and the heterogeneous distribution of reinforcing particles within the analyzed regions. Overall, the results confirm that mechanochemical processing ensures effective mixing of the starting powders and promotes a relatively homogeneous distribution of the vanadium-containing phase throughout the AlN matrix.

Table 2 – Average elemental composition of the (1-x)AlN-xVC composites determined by EDS analysis (mean ± standard deviation, n = 3)

Composition	C (wt.%)	N (wt.%)	Al (wt.%)	V (wt.%)
AlN (x = 0)	–	40.20 ± 0.44	59.80 ± 0.44	–
(1-x)AlN-xVC (x = 0.01)	0.50 ± 0.46	0.50 ± 0.46	0.50 ± 0.46	0.50 ± 0.46
(1-x)AlN-xVC (x = 0.03)	0.83 ± 1.38	0.83 ± 1.38	0.83 ± 1.38	0.83 ± 1.38
(1-x)AlN-xVC (x = 0.05)	3.07 ± 0.55	3.07 ± 0.55	3.07 ± 0.55	3.07 ± 0.55
(1-x)AlN-xVC (x = 0.10)	6.47 ± 1.59	35.80 ± 1.05	49.60 ± 1.74	8.13 ± 1.20

Figure 5 presents the X-ray diffraction patterns of the (1-x)AlN-xTaC composites after mechanochemical activation followed by heat treatment. The diffraction patterns are dominated by reflections corresponding to the hexagonal AlN phase (space group $P6_3mc$), indicating that AlN remains the principal constituent of the composites over the entire investigated concentration range.

At low TaC contents (x = 0.01), no distinct reflections associated with tantalum carbide are observed, suggesting that the carbide content is below the detection limit of XRD or that the Ta-containing particles remain finely dispersed within the AlN matrix. With increasing TaC concentration (x ≥ 0.03), weak diffraction peaks attributable to TaC become visible and gradually increase in intensity, confirming the presence of the reinforcing carbide phase without the formation of additional reaction products.



1) x = 0; 2) x = 0.01; 3) x = 0.03; 4) x = 0.05; 5) x = 0.10

Figure 5 – X-ray diffraction patterns of the (1-x)AlN-xTaC composites after mechanochemical activation and heat treatment

The absence of detectable secondary oxide phases indicates that TaC remains thermodynamically stable under the applied heat-treatment conditions. This behavior is consistent with the exceptionally high chemical and oxidation stability of tantalum carbide, which belongs to the family of ultra-high-temperature ceramics and exhibits excellent resistance to oxidation compared with many other transition-metal carbides.

The calculated lattice parameters of the AlN phase (Table 3) show only minor variations with increasing TaC content. Both the a and c lattice parameters remain relatively stable over the investigated composition range, indicating that the crystal structure of the AlN matrix is largely preserved after mechanochemical processing and subsequent heat treatment. Considering the extremely limited mutual solubility between AlN and TaC, these slight variations are more likely associated with residual microstrains and lattice distortions introduced during high-energy milling than with substitutional incorporation of tantalum atoms into the AlN lattice. The absence of significant changes in the lattice parameters is also consistent with the XRD results, which demonstrate that TaC is retained as a separate reinforcing phase rather than forming a solid solution with AlN. These observations indicate that the strengthening effect of TaC primarily originates from its role as a dispersed reinforcing phase and from the microstructural refinement induced by mechanochemical processing, rather than from modifications of the AlN crystal lattice.

Table 3 – Structural parameters of the (1-x)AlN-xTaC composites determined from XRD analysis

Phase	x=0.0	x=0.01	x=0.03	x=0.05	x=0.10
AlN (hexagonal, P63mc)	a=3.1192 c=4.9906 V=42.05	a=3.1105 c=4.9819 V=41.74	a=3.1112 c=4.9819 V=41.76	a=3.1144 c=4.9829 V=41.86	a=3.1149 c=4.9819 V=41.86
TaC (cubic, Fm-3m)	-	a=4.4572 V=88.55	a=4.4565 V=88.51	a=4.4574 V=88.56	a=4.4573 V=88.55
Degree of structural ordering, %	89.5	89.2	88.1	86.9	86.2

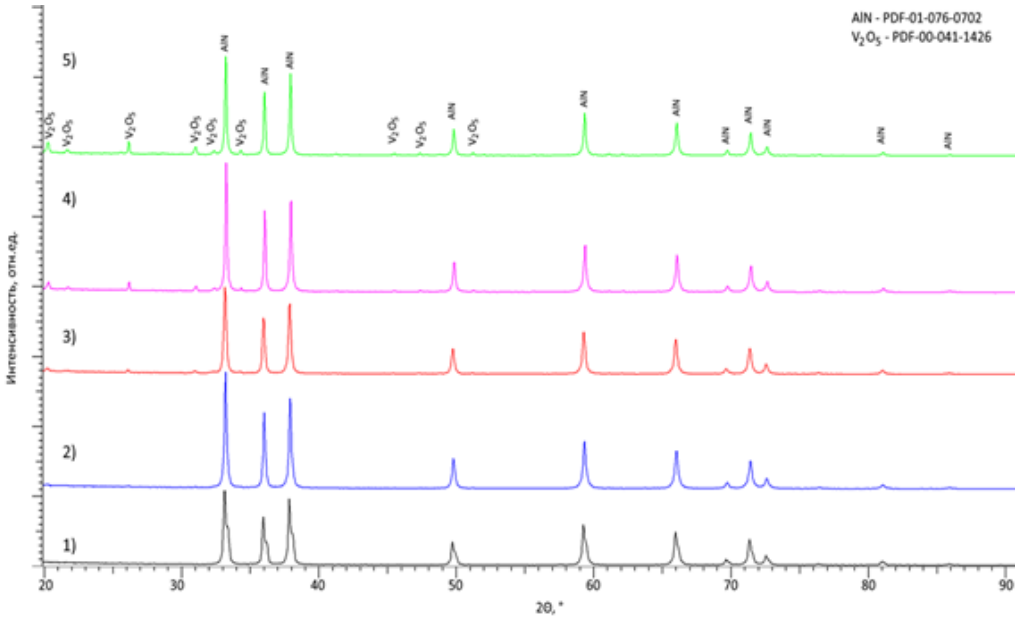
Overall, the XRD results indicate that TaC remains chemically stable during mechanochemical processing and subsequent heat treatment and is retained as a distinct reinforcing phase. No oxide phases were detected, indicating that the selected processing conditions preserve the integrity of the carbide. Combined with the homogeneous particle distribution observed by SEM/EDS analysis, the retained TaC phase provides favorable conditions for effective dispersion strengthening, grain-boundary pinning, and inhibition of grain growth, thereby contributing to the improved mechanical properties of the AlN-TaC composites.

Figure 6 presents the X-ray diffraction patterns of the (1-x)AlN-xVC composites after heat treatment. In contrast to the TaC-containing samples, the phase evolution of the VC-modified composites differs substantially.

The diffraction patterns remain dominated by the hexagonal AlN phase; however, beginning at x = 0.03, additional reflections corresponding to orthorhombic V_2O_5 become apparent. The intensity of these reflections increases progressively with increasing VC content, whereas characteristic peaks of crystalline VC are absent throughout the investigated composition range.

The disappearance of VC reflections together with the appearance of V_2O_5 indicates that vanadium carbide undergoes oxidation during heat treatment in air. The oxidation behavior can be attributed to the comparatively lower thermodynamic stability and oxidation resistance of VC relative to TaC. Under the present processing conditions, oxygen supplied by the annealing atmosphere reacts preferentially with VC, leading to the formation of vanadium oxide rather than preserving the original carbide phase. Similar oxidation behavior of vanadium-containing carbides has been reported previously in the literature.

The structural parameters summarized in Table 4 reveal only slight changes in the lattice constants of the AlN phase, whereas the degree of structural ordering gradually decreases with increasing VC concentration. This trend suggests that the formation of V_2O_5 and the associated lattice distortions introduce additional structural heterogeneity into the composite. The observed decrease in structural ordering is therefore attributed primarily to residual lattice strain and local structural distortions generated during mechanochemical processing and subsequent oxidation rather than to significant changes in the AlN crystal structure itself.



1) x = 0; 2) x = 0.01; 3) x = 0.03; 4) x = 0.05; 5) x = 0.10

Figure 6 – X-ray diffraction patterns of the (1-x)AlN – xVC composites after mechanochemical activation and heat treatment

Table 4 – Structural parameters of the (1-x)AlN-xVC composites determined from XRD analysis

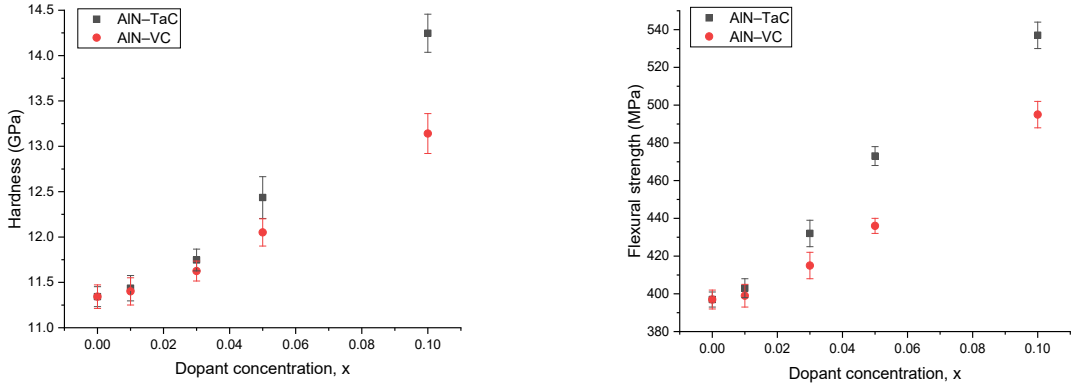
Phase	x=0.0	x=0.01	x=0.03	x=0.05	x=0.10
AlN (hexagonal, P63mc)	a=3.1192 c=4.9906 V=42.05	a=3.1131 c=4.9827 V=41.82	a=3.1179 c=4.9886 V=42.01	a=3.1058 c=4.9752 V=41.56	a=3.1106 c=4.9781 V=41.71
V2O5 (orthorhombic, Pmmn(59))	-	-	a=11.5410 b=3.5684 c=4.3762 V=180.23	a=11.4912 b=3.558 c=4.3667 V=178.54	a=11.4641 b=3.5622 c=4.3754 V=178.68
Degree of structural ordering, %	89.5	89.3	89.4	88.4	87.9

In contrast to the TaC-containing composites, the XRD results demonstrate that VC undergoes partial oxidation during heat treatment, leading to the formation of the V_2O_5 phase at $x \geq 0.03$. No diffraction peaks corresponding to crystalline VC are detected after thermal treatment, suggesting that a substantial fraction of the initial carbide is transformed into vanadium oxide under the selected processing conditions. This behavior is consistent with the comparatively lower oxidation resistance and thermodynamic stability of VC relative to TaC.

The formation of V_2O_5 is also consistent with the SEM observations (Figure 3), where finely dispersed oxide-rich regions are observed in the vicinity of particle agglomerates. Together, the SEM and XRD results indicate that oxidation of VC plays an important role in determining the final phase composition and microstructural evolution of the composites.

The different phase evolution observed for the TaC- and VC-containing systems is expected to influence their strengthening behavior. Whereas TaC remains chemically stable and is retained as a reinforcing carbide phase, partial oxidation of VC reduces the amount of intact carbide available for reinforcement and introduces secondary oxide phases into the microstructure. These differences provide a plausible explanation for the different mechanical responses of the investigated composites, which are discussed in the following section.

Figure 7 summarizes the variation in the Vickers hardness and flexural strength of the (1-x)AlN-xTaC and (1-x)AlN-xVC composites as a function of carbide content. Both mechanical properties increase with increasing dopant concentration; however, the magnitude of the improvement differs considerably for the two reinforcing carbides.



a) b)

a) Vickers hardness; b) flexural strength

Figure 7 – Mechanical properties of the (1-x)AlN-xTaC and (1-x)AlN-xVC composites as a function of carbide content

At low additive contents ($x \leq 0.03$), only a moderate improvement in hardness and flexural strength is observed for both systems, suggesting that the volume fraction of the reinforcing phase is insufficient to produce a pronounced strengthening effect. As the carbide content increases to $x = 0.05$ and 0.10, both mechanical properties increase more noticeably, indicating that the reinforcing particles become sufficiently abundant to effectively impede deformation and crack propagation.

The strengthening effect is consistently more pronounced for the TaC-containing composites than for the VC-containing materials. The hardness of the AlN-TaC composites increases by approximately 10–25% relative to monolithic AlN, whereas the corresponding increase for the AlN-VC system is limited to approximately 6–15%. A similar tendency is observed for flexural strength.

The superior mechanical performance of the TaC-containing composites can be explained by the combined effects of phase stability, microstructural evolution, and several complementary strengthening mechanisms. As demonstrated by the XRD results (Figure 4), TaC remains chemically stable during mechanochemical processing and subsequent heat treatment, preserving the hard carbide phase within the AlN matrix. SEM observations (Figure 1) and EDS elemental mapping (Figure 2) further confirm the relatively homogeneous distribution of TaC particles without pronounced agglomeration.

Uniformly dispersed TaC particles act as effective obstacles to localized deformation, providing dispersion strengthening. In addition, carbide particles located at grain boundaries suppress grain growth during thermal treatment, thereby contributing to grain-boundary strengthening according to the Hall-Petch relationship. Although quantitative grain-size measurements were not performed, the improved powder homogeneity observed after mechanochemical processing suggests that dispersed carbide particles restrict grain coarsening during subsequent heat treatment. Furthermore, the uniformly distributed hard particles promote crack deflection and crack bridging, thereby increasing the energy required for crack propagation and contributing to the observed improvement in flexural strength. These strengthening mechanisms operate synergistically with the refined microstructure produced by mechanochemical activation, resulting in superior mechanical performance.

In contrast, the strengthening efficiency of VC is lower because the reinforcing phase undergoes partial oxidation during heat treatment. The XRD analysis (Figure 6) demonstrates the formation of V_2O_5 at $x \geq 0.03$, while no crystalline VC reflections are detected after processing. Consequently, part of the initially introduced carbide is converted into an oxide phase possessing lower hardness and lower reinforcing capability than the original carbide. This phase transformation reduces the effective volume fraction of hard reinforcing particles available for load transfer and limits the overall strengthening effect. The presence of oxide-rich regions identified by SEM and EDS observations further supports this interpretation.

Overall, the obtained results demonstrate that the mechanical behavior of the investigated composites is governed not only by the amount of reinforcing additive but also by its chemical stability during processing and the resulting phase composition. The superior performance of the AlN-TaC composites arises from the retention of a stable carbide phase, its homogeneous dispersion within the AlN matrix, and the combined contributions of dispersion strengthening, grain-growth inhibition, grain-boundary strengthening, and crack-deflection mechanisms. In contrast, the partial oxidation of VC into V_2O_5 reduces the efficiency of these strengthening mechanisms, leading to comparatively lower hardness and flexural strength. These observations establish a clear relationship between phase evolution, microstructure, and mechanical performance, thereby providing a mechanistic explanation for the different responses of the TaC- and VC-reinforced AlN composites.

Conclusion

This study systematically investigated the influence of tantalum carbide and vanadium carbide additions on the phase composition, microstructure, and mechanical properties of AlN-based ceramic composites prepared by mechanochemical solid-state processing.

The results demonstrate that mechanochemical activation provides effective homogenization of the powder mixtures and promotes a uniform distribution of the reinforcing phases prior to heat treatment. The AlN-TaC composites retain a stable carbide phase after processing, whereas the AlN-VC system undergoes partial oxidation, resulting in the formation of V_2O_5. These differences in phase evolution significantly influence the subsequent microstructural development and mechanical behavior of the composites.

Mechanical characterization showed that increasing the carbide content above $x=0.03$ leads to a noticeable improvement in hardness and flexural strength. The strengthening effect is more pronounced for the TaC-containing composites, which can be attributed to the retention of a chemically stable, uniformly dispersed hard carbide phase. In contrast, partial oxidation of VC reduces the amount of the reinforcing carbide available for strengthening and consequently limits the improvement in mechanical performance.

The combined SEM, EDS, XRD, and mechanical characterization establishes a clear relationship between dopant type, phase evolution, microstructure, and mechanical properties. The observed strengthening is associated with the combined effects of homogeneous dispersion of the reinforcing phase, inhibition of excessive grain growth, grain-boundary strengthening, and increased resistance to crack propagation.

Although thermal conductivity was not investigated in the present work, the obtained results indicate that TaC is a promising reinforcing additive for improving the mechanical reliability of AlN-based ceramics while maintaining a stable phase composition under the applied processing conditions.

The developed AlN-based composites may therefore be considered promising candidates for structural ceramic components operating under elevated temperatures and severe mechanical loading, including wear-resistant components, high-temperature structural elements, protective ceramic parts, and thermally stable substrates where enhanced mechanical reliability is required. The established composition-structure-property relationships may also provide useful guidance for the further design and optimization of advanced AlN-based ceramic composites.

Funding information. This research was funded by the Science Committee of the Ministry of Education and Science of the Republic of Kazakhstan (No. BR28713365).

REFERENCES

1 Schwarzer-Fischer, E., Scheithauer, U., and Michaelis, A. CerAMfacturing of aluminum nitride with high thermal conductivity via lithography-based ceramic vat photopolymerization (CerAM VPP). Ceramics, 6 (1), 416–431 (2023). https://doi.org/10.3390/ceramics6010024

2 Gaskins, J.T., Hopkins, P.E., Merrill, D.R., Bauers, S.R., Hadland, E., Johnson, D.C., and King, S.W. Investigation and review of the thermal, mechanical, electrical, optical, and structural properties of atomic layer deposited high-k dielectrics: Beryllium oxide, aluminum oxide, hafnium oxide, and aluminum nitride. ECS Journal of Solid State Science and Technology, 6 (10), N189 (2017). https://doi.org/10.1149/2.0091710jss

3 Zhang, Z., Wu, H., Zhang, Z., Li, J., Xu, H., Lu, H., and Qin, M. Limiting the lattice oxygen impurities to obtain high thermal conductivity aluminum nitride ceramics. Ceramics International, 50 (18), 33488–33495 (2024). https://doi.org/10.1016/j.ceramint.2024.06.164

4 Zhang, Z., Wu, H., Li, T., Zhang, Z., Xu, H., Lu, H., and Qin, M. Preparation of high thermal conductivity aluminium nitride ceramics with low oxygen impurity for power semiconductors. Electrical Materials and Applications, 1 (1), e12012 (2024). https://doi.org/10.1049/ema3.12012

5 Kozlovskiy, A.L., Borgekov, D.B., Tleulessova, I.K., Zhumazhanova, A.T., Moldabayeva, G.Z., Burkhanov, B.Z., and Khametova, A.A. Evaluation of the application efficiency of multicomponent ceramics as protective shielding and thermal barrier materials. Eurasian Physical Technical Journal, 22 (3), 24–35 (2025). https://doi.org/10.31489/2025N3/24-35

6 Paik, U., Park, H.C., Choi, S.C., Ha, C.G., Kim, J.W., and Jung, Y.G. Effect of particle dispersion on microstructure and strength of reaction-bonded silicon carbide. Materials Science and Engineering: A, 334 (1-2), 267–274 (2002). https://doi.org/10.1016/S0921-5093(01)01897-4

7 Mao, H., Shen, F., Zhang, Y., Wang, J., Cui, K., Wang, H., and Tan, T. Microstructure and mechanical properties of carbide reinforced TiC-based ultra-high temperature ceramics: A Review. Coatings, 11 (12), 1444 (2021). https://doi.org/10.3390/coatings11121444

8 Yue, X.M., Zhang, G.J., and Wang, Y.M. Reaction synthesis and mechanical properties of TiB2–AlN–SiC composites. Journal of the European Ceramic Society, 19 (3), 293–298 (1999). https://doi.org/10.1016/S0955-2219(98)00150-2

9 Lin, K., Zong, X., Sheng, P., Zhao, S., Qi, Y., Zhang, W., and Wu, S. Effects of SmF3 addition on aluminum nitride ceramics via pressureless sintering. Journal of the European Ceramic Society, 43 (15), 6804–6814 (2023). https://doi.org/10.1016/j.jeurceramsoc.2023.07.051

10 Mao, H., Zhang, Y., Wang, J., et al. Microstructure, Mechanical Properties, and Reinforcement Mechanism of Second-Phase Reinforced TiC-Based Composites: A Review. Coatings, 12 (6), 801 (2022). https://doi.org/10.3390/coatings12060801

11 Yang, S.L., Chu, C.H., and Hsiang, H.I. Effect of Y2Zr2O7 content on improving mechanical properties and thermal shock resistance of AlN-based composite ceramics. Ceramics International, 50, 29321–29331 (2024). https://doi.org/10.1016/j.ceramint.2024.05.226

12 Zhai, X., Lu, K., Peng, C., et al. Effects of Nb/TiC/TaC/VC and Co addition on the microstructure and properties of WC-based cemented carbides. International Journal of Applied Ceramic Technology, 20, 2785–2795 (2023). https://doi.org/10.1111/ijac.14424

13 Sun, W., Kuang, X., Liang, H., Xia, X., Zhang, Z., Lu, C., and Hermann, A. Mechanical properties of tantalum carbide from high-pressure/high-temperature synthesis and first-principles calculations. Physical Chemistry Chemical Physics, 22 (9), 5018–5023 (2020). https://doi.org/10.1039/C9CP06819H

14 Peng, J., Dong, H., Hojamberdiev, M., Yi, D., Yang, Y., Bao, H., and Meng, L. Improving the mechanical properties of tantalum carbide particle-reinforced iron-based composite by varying the TaC contents. Journal of Alloys and Compounds, 726, 896–905 (2017). https://doi.org/10.1016/j.jallcom.2017.08.050

15 Fahrenholtz, W.G., and Hilmas, G.E. Ultra-high temperature ceramics: materials for extreme environments. Scripta Materialia, 129, 94–99 (2017). https://doi.org/10.1016/j.scriptamat.2016.10.018

16 Zeiger, M., Ariyanto, T., Krüner, B., Peter, N.J., Fleischmann, S., Etzold, B.J., and Presser, V. Vanadium pentoxide/carbide-derived carbon core–shell hybrid particles for high performance electrochemical energy storage. Journal of Materials Chemistry A, 4 (48), 18899–18909 (2016). https://doi.org/10.1039/C6TA08900C

17 Bugdayci, M., Once, L., Alkan, M., Turan, A., and Cinarli, U. A Comparative Study about Production of Vanadium Carbide via Self Propagating High Temperature Synthesis and Reduction. Archives of Metallurgy and Materials, 69 (1), 257–262 (2024). https://doi.org/10.24425/amm.2024.147816

18 Baláž, P. Mechanochemistry in minerals engineering. Mechanochemistry in Nanoscience and Minerals Engineering (Berlin, Heidelberg: Springer Berlin Heidelberg, 2008), pp. 257–296.

19 Sritharan, T., Boey, F.Y.C., and Srinivas, A. Synthesis of complex ceramics by mechanochemical activation. Journal of Materials Processing Technology, 192, 255–258 (2007). https://doi.org/10.1016/j.jmatprotec.2007.04.079

20 Maghsoudipour, A., Moztarzadeh, F., Saremi, M., and Heinrich, J.G. Oxidation behavior of AlN–Al2O3 composites. Ceramics International, 30 (5), 773–783 (2004). https://doi.org/10.1016/j.ceramint.2003.10.004

21 Varanasi, D., Furkó, M., Balázsi, K., and Balázsi, C. Processing of Al2O3-AlN ceramics and their structural, mechanical, and tribological characterization. Materials, 14 (20), 6055 (2021). https://doi.org/10.3390/ma14206055

[1*]Жумажанова А.Т.,
PhD, КҒҚ, ORCID ID: 0000-0002-5483-9552,
* e-mail: ainashzhumazhanova@gmail.com
[1]Шакирзянов Р.И.,
ф.-м. ғ.к., КҒҚ, ORCID ID: 0000-0001-9908-3034,
e-mail: shakirzyanov_ri@enu.kz

[1]Л.Н. Гумилев атындағы Еуразия ұлттық университеті, Астана қ., Қазақстан

ТАНТАЛ ЖӘНЕ ВАНАДИЙ КАРБИДТІ ДОПАНТТАРЫНЫҢ АЛЮМИНИЙ НИТРИДІ НЕГІЗІНДЕГІ КЕРАМИКАЛАРДЫҢ ҚҰРЫЛЫМЫ МЕН МЕХАНИКАЛЫҚ ҚАСИЕТТЕРІНЕ ӘСЕРІ

Аңдатпа

Бұл жұмыста механохимиялық қатты фазалық өңдеу және кейінгі термиялық өңдеу арқылы алынған алюминий нитриді негізіндегі керамикалық композиттердің фазалық құрамына, микроқұрылымына және механикалық қасиеттеріне тантал карбиді мен ванадий карбиді қоспаларының әсері зерттелді. Карбидтік қоспалардың мольдік үлесі x=0,01–0,10 аралығында өзгертілді. Сканерлеуші электрондық микроскопия және энергодисперсиялық рентгендік спектрлік талдау нәтижелері механохимиялық активациядан кейін арматуралаушы фазалардың біркелкі таралатынын көрсетті. Рентгендік фазалық талдау TaC өңдеу барысында химиялық тұрақтылығын сақтап, AlN матрицасының кристалдық торындағы деформацияларды азайта отырып, екі фазалы AlN-TaC композитінің түзілетінін көрсетті. Керісінше, VC термиялық өңдеу кезінде ішінара тотығып, екінші реттік V_2O_5 фазасының түзілуіне алып келеді, нәтижесінде материалдың фазалық эволюциясы өзгеше сипат алады. Механохимиялық өңдеу ұнтақ қоспасының анағұрлым біртекті морфологиясының қалыптасуына ықпал етіп, карбид бөлшектерінің ірі агломераттарының түзілуін тежейді, бұл кейінгі микроқұрылымның қалыптасуына қолайлы жағдай жасайды. Механикалық сынақтар $x > 0,03$ болған жағдайда қаттылық пен иілу кезіндегі беріктіктің едәуір артатынын көрсетті. Ең жоғары беріктендіру әсері тұрақты, біркелкі таралған жоғары қаттылықтағы карбидтік фазаның сақталуына байланысты TaC қосылған композиттерде байқалды. Механикалық қасиеттердің жақсаруы дисперсиялық беріктендірудің, түйіршіктердің өсуін тежеудің, түйіршік шекараларының беріктенуінің және жарықшақтардың таралу траекториясының ауытқуының бірлескен әсерімен түсіндіріледі. Алынған нәтижелер өңдеу шарттары, фазалық эволюция, микроқұрылым және механикалық қасиеттер арасындағы өзара байланысты анық көрсетіп, жоғары механикалық және термиялық жүктемелер жағдайында пайдалануға арналған алюминий нитриді негізіндегі жоғары тиімді керамикалық композиттерді әзірлеуде механохимиялық өңдеу әдісінің перспективалы екенін дәлелдейді.

Түйін сөздер: алюминий нитриді, карбо-нитридті керамикалар, механохимиялық синтез, тантал карбиді, ванадий карбиді, фазалық құрам, беріктік қасиеттері.

[1*]Жумажанова А.Т.,
PhD, МНС, ORCID ID: 0000-0002-5483-9552,
* e-mail: ainashzhumazhanova@gmail.com
[1]Шакирзянов Р.И.,
к.ф.-м.н., МНС, ORCID ID: 0000-0001-9908-3034,
e-mail: shakirzyanov_ri@enu.kz

[1]Евразийский национальный университет им. Л.Н. Гумилева, г. Астана, Казахстан

ВЛИЯНИЕ КАРБИДНЫХ ДОПАНТОВ ТАНТАЛА И ВАНАДИЯ НА СТРУКТУРУ И МЕХАНИЧЕСКИЕ СВОЙСТВА КЕРАМИК НА ОСНОВЕ НИТРИДА АЛЮМИНИЯ

Абстракт

В настоящей работе исследовано влияние добавок карбида тантала и карбида ванадия на фазовый состав, микроструктуру и механические свойства керамических композитов на основе нитрида алюминия, полученных методом механохимической твердофазной обработки с последующей термообработкой. Мольная доля карбидных добавок варьировалась в диапазоне x = 0,01–0,10. Результаты сканирующей электронной микроскопии в сочетании с энергодисперсионным рентгеноспектральным анализом показали однородное распределение армирующих фаз после механохимической активации. Рентгенофазовый анализ показал, что карбид тантала сохраняет химическую стабильность в процессе обработки, что приводит к формированию двухфазного композита AlN-TaC с пониженными искажениями кристаллической решетки матрицы AlN. Напротив, карбид ванадия в процессе термообработки подвергается частичному окислению с образованием вторичной фазы V_2O_5, что обусловливает иной характер фазовой эволюции материала. Механохимическая обработка способствует формированию более однородной морфологии порошковой смеси и подавляет образование крупных агломератов карбидных частиц, создавая благоприятные условия для последующего формирования микроструктуры. Механические испытания показали, что при содержании добавок x > 0,03 наблюдается существенное повышение твердости и прочности при изгибе, при этом наибольший эффект упрочнения достигается в композитах, содержащих TaC, благодаря сохранению стабильной равномерно распределенной высокотвердой карбидной фазы. Улучшение механических свойств обусловлено совместным действием дисперсионного упрочнения, подавления роста зерен, упрочнения границ зерен и отклонения траектории распространения трещин. Полученные результаты устанавливают четкую взаимосвязь между условиями обработки, фазовой эволюцией, микроструктурой и механическими свойствами материалов, демонстрируя перспективность механохимического метода для создания высокоэффективных керамических композитов на основе нитрида алюминия, предназначенных для эксплуатации в условиях высоких механических и термических нагрузок.

Ключевые слова: нитрид алюминия, карбо-нитридные керамики, механохимический синтез, карбид тантала, карбид ванадия, фазовый состав, прочностные свойства.