



Features of Synthesis of a Metal-Matrix Composite Based on Natural Calcium Carbonate

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Abstract

This study investigates the possibility of using calcite-shell limestone, widely available in Tajikistan, as a filler for an aluminum matrix in the synthesis of a metal matrix composite. Using aluminum as the matrix enables the composite to be produced at a relatively low temperature (700 °C), avoiding the intense decomposition of calcium carbonate that begins at 800–900 °C. The structural and phase changes occurring in the material during the activation and modification of calcite were examined. It was shown that modifying the calcite surface enhances the interfacial interaction between the filler and the aluminum matrix, improving adhesion and the efficiency of mechanical stress transfer. It was established that a 10 mm-thick composite containing 20 wt.% aluminum and 80 wt.% calcite provided the greatest attenuation of gamma and X-ray radiation, reducing the X-ray dose rate from 1490 to 135 $\mu\text{Sv/h}$ (approximately 11-fold).

Keywords:

mineral; calcite; composite; aluminum matrix composite; calcite filler; metal matrix composite

1. Introduction

For many centuries, people have highly valued natural raw materials, among which calcium carbonate holds particular importance. Calcite is characterized by low water solubility, and its powdered form is widely used in cement production, one of the key components of masonry and plastering mortars [1,2].

Calcite contributes to the production of durable, long-lasting binding materials that retain their operational properties indoors and outdoors for many years [3,4].

The subsoil of Tajikistan contains significant reserves of natural minerals such as barite, calcite, and bentonite, among others, which possess promising physico-chemical properties for use in radiation-shielding composite materials [5].

In this context, scientific research aimed at identifying, developing, and scientifically justifying technologies for producing environmentally safe building materi-

als with radiation-protection properties, based on local natural resources, is becoming particularly relevant.

The synthesis of metal matrix composites using natural calcium carbonate is a promising direction that combines environmental friendliness with enhanced mechanical properties in metals. The introduction of calcium carbonate particles into the metal matrix exerts a strengthening effect, increasing hardness, yield strength, and tensile strength.

One of the main challenges in creating such composites remains porosity, along with insufficient wettability between the metal matrix and the ceramic or mineral particles. To address these issues, various material processing methods are employed, including surface activation and modification [6,7].

The most common and economically viable method for producing metal matrix composites is casting with mixing, in which natural calcite is introduced into the molten metal during mechanical mixing. A significant challenge in using calcium carbonate is its limited thermal stability:

at temperatures above 800–900 °C, calcium carbonate begins to decompose intensively, releasing carbon dioxide. The released gas can lead to the formation of pores and internal defects in the metal matrix. Accordingly, modern research places particular emphasis on developing protective coatings for calcium carbonate particles, as well as low-temperature sintering methods.

Aluminum is used as the matrix in these composite materials because of the relative stability of its physical and mechanical properties across a wide temperature range. This makes it possible to obtain composite materials with specified operational characteristics while optimizing production parameters and reducing environmental impact.

The aim of this study is to investigate the features of synthesizing a metal-matrix composite based on natural calcium carbonate, and to assess the impact of modified calcite on the structure, interfacial interaction, and radiation-protection properties of the aluminum matrix.

The choice of material is justified by the fact that calcite shell rock is a widespread and inexpensive natural material in Tajikistan. Its use as a filler in developing new radiation-shielding metal-matrix composites is promising from both economic and environmental perspectives.

2. Materials and Methods

2.1. Materials

For the synthesis of the composite material, natural calcium carbonate of local origin from deposits in Tajikistan was selected, represented by the sedimentary calcite mineral—oolitic limestone. As part of the study, 10–15 point samples were collected at each site, each consisting of three mini-samples weighing 200–300 g, taken from the upper and middle layers. The mini-samples were then combined into a single sample weighing 2–3 kg. Shell samples were pre-dried and ground to a fraction of less than 0.074 mm before analytical studies, ensuring the homogeneity of the material under investigation and increasing the reliability of the phase analysis results.

In the processes of activating and modifying the natural calcium carbonate, the following substances were used:

- $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ (GOST 450-77), manufacturer—“Prime ChemicalsGroup,” Russia;
- $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ (GOST 3759-75), manufacturer—LLC “Component-Reactive,” Russia.

2.1.1. Sample Preparation

In the first stage, aqueous solutions of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ (0.7 M) and $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ (0.1 M) were prepared.

To activate 10 g of calcite, it was mixed with 100 mL of water, after which 20 mL of a $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ (0.7 M) solution was added to the suspension, and mixing continued for 30 min. The material was then filtered and dried at a temperature of 80 °C.

For modification, 10 g of activated calcite was mixed with 100 mL of water and 30 mL of an $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ (0.1 M) solution. The resulting mixture was stirred with a mechanical stirrer for 30 min, after which the material was washed and dried at a temperature of 120–160 °C.

2.2. Methods

2.2.1. Synthesis of a Composite

Four metal-matrix composite samples were prepared with the compositions shown below in Table 1:

During synthesis of the composite, metallic aluminum of grade A0 (GOST 11069-2001/2019), produced by TALCO, Tajikistan, was used as the matrix.

Metallic aluminum was melted at a temperature of 700 °C, followed by the introduction of modified calcite filler with intensive mechanical mixing until a homogeneous composite mixture was obtained. The resulting mixture was poured into a mold and heated in an oven at 700 °C for 30 min. The molds were then removed from the furnace and subjected to hot pressing at a pressure of 100 MPa, yielding an aluminum matrix composite.

2.2.2. Role of Components in the Composite

- Calcite (filler)—contributes to the reduction of gamma and X-ray radiation intensity;
- $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ (activator)—activates the surface of calcite;
- $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ (modifier)—modifies the surface of calcite;
- Metallic aluminum (matrix)—imparts lightness, plasticity, non-toxicity, and corrosion resistance to the composite material.

2.2.3. X-Ray Phase Analysis

The phase composition of the samples was determined using X-ray phase analysis with an XRDynamic 500 X-ray diffractometer produced by Anton Paar (Austria). This method enables reliable identification of the crystalline phases of the material under investigation, based on analysis of the diffraction maxima recorded as a function of the diffraction angle 2θ .

Differential thermal analysis was used to investigate the thermal transformations of the initial mineral raw material. Thermal studies were conducted using a Labsys Evo-C1600 derivatograph manufactured by Setaram Instrumentation (France). This method enables determination of the temperature ranges of phase and chemical transformations accompanied by changes in mass and thermal effects.

The surface morphology and microstructure of the particles were examined using a JEOL JSM-IT200 scanning electron microscope (Japan).

The radiation-protection properties of the composites were tested using the following gamma radiation sources:

- ^{60}Co ($E = 1.25 \text{ MeV}$);

- ^{133}Ba ($E = 0.35 \text{ MeV}$);
- dosimeter ESMFH40G-L10 THERMO (Germany), calibration—February 2026.

The samples were positioned 30 cm from the source, at the center of a 2 cm diameter gamma radiation beam.

The attenuation of the X-ray dose rate was determined using an SF-80 X-ray machine (Japan) and a DKS AT 1121 dosimeter (Republic of Belarus), with the equipment configured to exclude scattered radiation.

2.2.4. Testing Conditions

- distance to the source—1 m;
- positioning—lateral;
- X-ray tube parameters: 70 kV, 200 mA, 0.080 s, 16 mAs.

Table 1: Composition of metal-matrix composites.

	Sample 1	Sample 2	Sample 3	Sample 4
Technical aluminum, wt.%	50	40	30	20
Modified calcite, wt.%	50	60	70	80

3. Results

The results of the X-ray phase analysis showed that the dominant crystalline phase of the studied sample is calcite (CaCO_3), with a mass fraction of 91.3%. This phase produces the most intense diffraction peaks, forming the main part of the X-ray diffraction pattern (Figure 1), indicating its predominant presence in the material's structure.

Zinc oxide (ZnO) was identified as a secondary phase in the sample, with a content of approximately 6.3% by mass. The presence of this phase is confirmed by diffraction peaks of medium intensity.

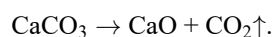
In addition, a small amount of rutile (TiO_2 ; approximately 2.4%) was detected, as indicated by low-intensity diffraction peaks.

The distribution and position of the diffraction maxima in the X-ray diffraction pattern are in good agreement with reference data for the identified crystalline phases. These results indicate a pronounced dominance of the calcite phase in the mineral structure of the shell rock. Comparison of the phase composition with known data on the chemical composition of shell limestone shows close agreement with the composition of calcite, which is characterized by approximately 56% CaO and 44.0% CO_2 .

Figure 2 presents the thermal curves of the calcite shell, showing the dependence of sample mass change (TG curve) and thermal effects (DTA curve) on heating temperature.

Analysis of the thermograms reveals a pronounced endothermic effect at a temperature of approximately 847.2°C , accompanied by a mass loss of 44.626 mg. This effect is associated with the thermal decomposition of the carbonate phase and the release of carbon dioxide. The thermal effect of this process is estimated at approximately $1.99 \times 10^5 \text{ J/mol}$, indicating intense thermochemical transformations in the structure of the calcite raw material.

These results confirm data from previously published studies [8]. The decomposition of calcium carbonate occurs in a single stage and is accompanied by the release of carbon dioxide, according to the reaction:



This process occurs at $800\text{--}900^\circ\text{C}$. Because the composite was synthesized at 700°C , below this decomposition range, calcium carbonate was retained as the filler without undergoing thermal decomposition.

Aluminum metal-matrix composites are widely used because of their low density and favorable performance properties [9].

Following the thermal studies, the calcite shell was subjected to an activation process to enhance the reactivity of its surface. Activation was achieved by hydroxylating the external surface of the particles in the presence of calcium ions adsorbed from the calcium chloride solution.

The pre-activated calcite was subsequently modified through adsorptive fixation of ions on the particle surface, using an aqueous solution of aluminum chloride.

This modification process occurs through electrostatic interaction between the charged active sites of the particles and the ions of the modifying agent, as well as

through ion-dipole interaction with the oxygen atoms of the hydroxyl groups present on the surface of the activated calcite particles. Notably, the high defectiveness of the calcite particle surfaces, together with a developed system of active centers, creates favorable conditions for the adsorptive fixation of the modifying components (Figure 3).

#	Phase ID	Chemical Formula	PDF-#	RiR	Wt%	BR%	FOM (n)	Scale(I)	Shift(x)	Space Group	a (Å)	b (Å)	c (Å)
1	Calcite	CaCO ₃	98-000-0141	2.91	91.3	8.5	5.1 (20)	0.592	0.020°	hR-3c (167)	4.9867	4.9867	17.0700
2	Zincite	ZnO	98-000-0483	5.35	6.3	10.0	9.4 (08)	0.026	0.080°	hP63mc (186)	3.2500	3.2500	5.2049
3	Rutile	TiO ₂	98-000-0375	3.31	2.4	8.5	18.0 (03)	0.024	0.060°	P42/mnm (136)	4.5032	4.5032	2.9620

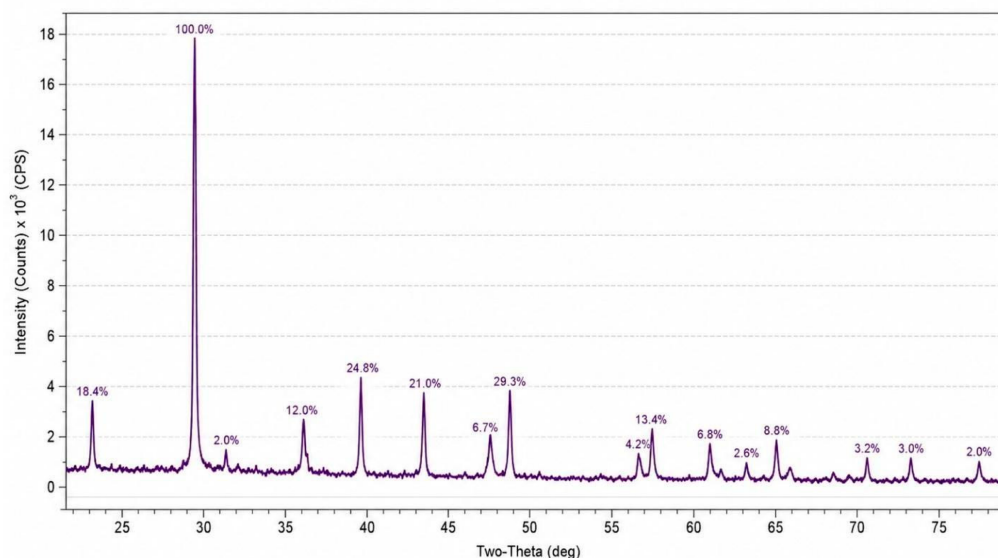


Figure 1: X-ray diffraction pattern of calcite shell rock.

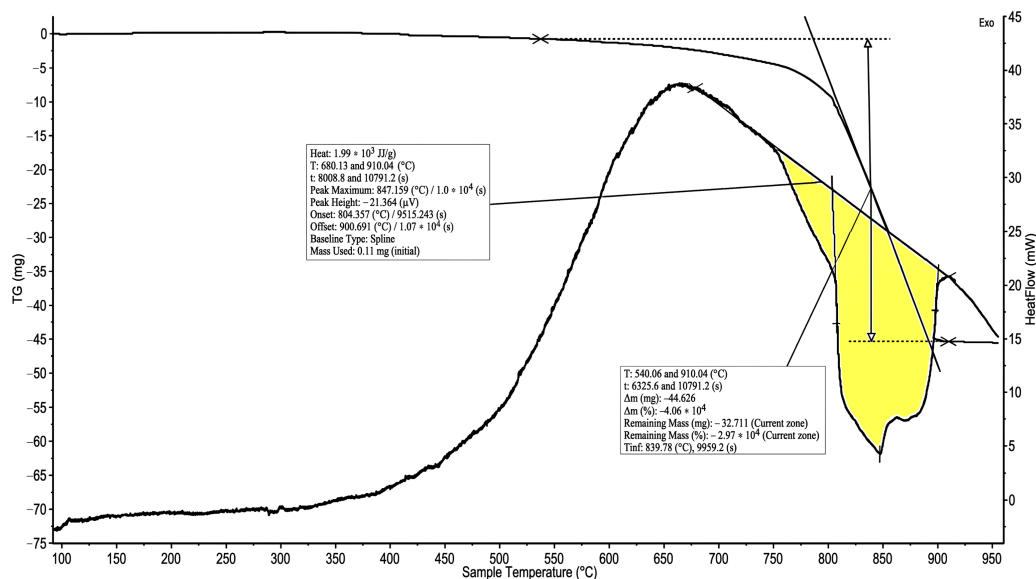


Figure 2: Thermal curves of calcite-limestone.

The calcite particles exhibit irregular morphology, agglomeration, and pronounced surface roughness. These surface features may promote stronger interfacial bonding with the aluminum matrix after modification.

Comparison of the calcite particle surfaces before and after modification reveals a metallic luster on the mod-

ified particles, which may be associated with an adsorbed aluminum-containing surface layer.

Surface modification increases the number of functional groups and promotes formation of a more developed interfacial layer, contributing to improved interaction between the mineral filler and the metal matrix (**Figure 4**).

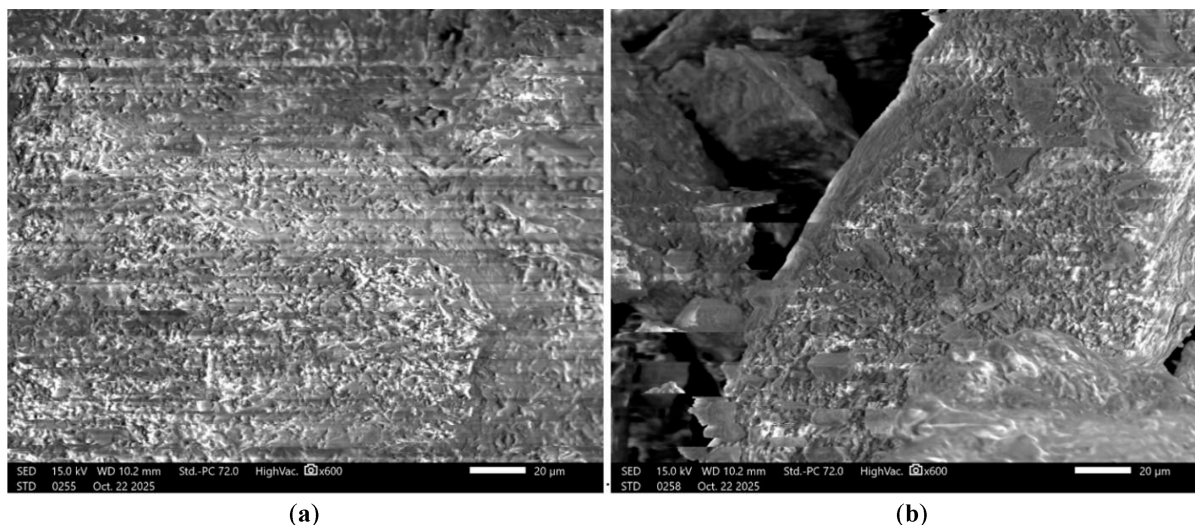


Figure 3: Microstructure of calcite: (a)—unmodified; (b)—modified.

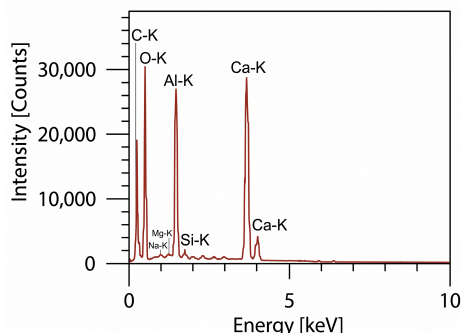


Figure 4: Spectra of modified calcite.

X-ray fluorescence analysis of the modified calcite shows a carbon content of 11.21 wt.%, oxygen 47.91 wt.%, calcium 39.41 wt.%, with the remainder consisting of impurities of Al, Si, Na, and Mg.

4. Discussion

The dominance of the calcite phase indicates the high carbonate character of the studied raw material, which can positively influence the structural formation of composite building materials and their physical and mechanical properties.

The presence of minor amounts of zinc oxide and rutile may further influence the microstructure and functional characteristics of the material, particularly its density, resistance to external influences, and potential radiation-protection properties. The established phase composition thus confirms the feasibility of using shell limestone as a mineral component in developing special-purpose construction materials, including environmentally safe radiation-protection compositions.

The modified calcite obtained was used as a dispersive filler in producing the metal-matrix composite. A key stage in the technology for producing metal matrix composite materials is the uniform distribution of filler particles within the metal matrix, since this factor largely determines the material's density, structural uniformity, mechanical properties, and functional characteristics. In forming the aluminum matrix composite, particular attention was given to ensuring the uniform distribution of modified calcite particles within the aluminum melt. The technological process involved melting aluminum at approximately 700 °C, followed by introduction of the modified calcite filler with intensive mechanical stirring until a homogeneous composite mixture was obtained (**Figure 5**).

After obtaining the composite melt, the material was placed in a mold and subjected to thermal treatment at 700 °C for 30 min. The samples were then pressed un-

der a pressure of 100 MPa, yielding metal-matrix composite materials in the form of 10 mm thick plates, intended

for subsequent studies, including the assessment of their radiation-protection properties.

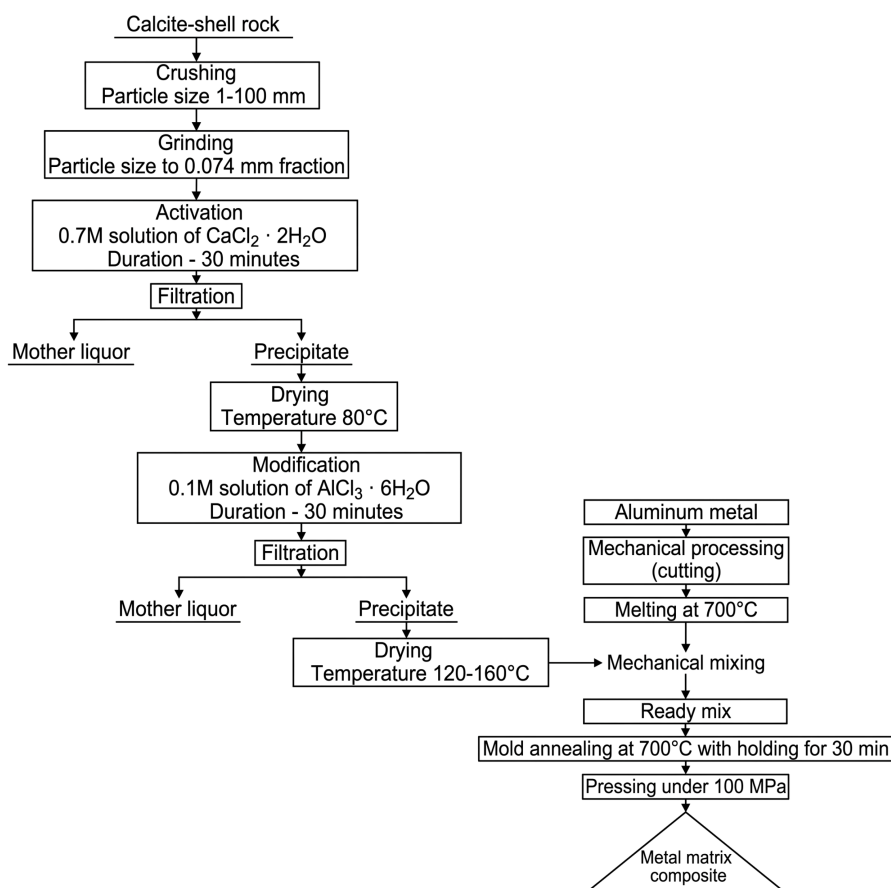


Figure 5: Technological scheme for obtaining a metal-matrix composite material based on calcite shell rock.

Earlier work by Varun Singhal et al. (2025) focused primarily on aluminum-based metal matrix composites, analyzing their properties, the influence of aging processes, and the potential for predictive modeling, thereby broadening the understanding of the prospects for these composites and their role in modern materials science [10].

M.S. Ayar et al. (2021) examined the current state of development of aluminum metal matrix composites, emphasizing their ability to conserve material and energy resources. The paper discusses key technological challenges related to the uniform distribution of the reinforcing phase and the high cost of processing, while noting significant prospects for the further development and adoption of these composites in engineering practice [11].

Sayed Ali Ahmad Alem et al. (2025) examined metal matrix composites reinforced with metallic particles, noting that such reinforcing materials possess a

unique combination of physical and mechanical properties that enhances the operational characteristics of the composites. Compared to traditional ceramic reinforcing materials, metallic particles demonstrate higher compatibility with metallic matrices. This work pays particular attention to the interaction mechanisms at the interface between the matrix and reinforcing phase, as well as the technological processes required to produce highly efficient metal matrix composites [12].

In [13], aluminum was employed as the matrix material for fabricating a radiation-shielding composite. It was established that the interfacial interaction between the aluminum matrix and finely dispersed hematite is facilitated by an aluminum oxide film formed on the surface of the filler particles.

In the present study, a similar interfacial mechanism is considered for the aluminum–modified calcium carbonate system. Owing to the structural compatibility of the

components, formation of a stable interfacial region is expected, enabling synthesis of the composite at a relatively low temperature of 700 °C without decomposition or loss of the calcite filler. Analysis of the microstructure of the

prepared aluminum-matrix composites showed a uniform distribution of aluminum throughout the composite volume (Figure 6).

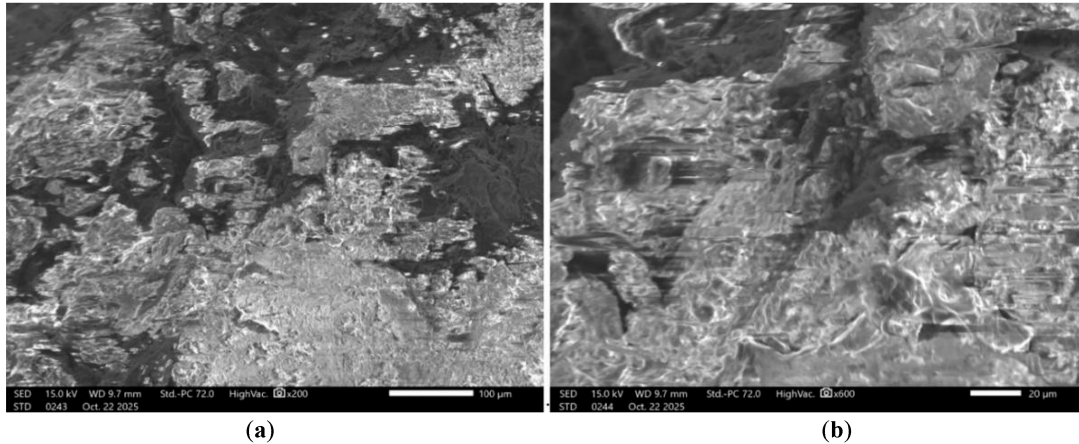


Figure 6: Microstructure of the aluminum-matrix composite at (a) $\times 200$ and (b) $\times 600$ magnification.

At high magnification, the particles are positioned close to one another, and the interparticle spaces are filled with aluminum. This morphology indicates good interfacial contact between the calcite particles and the aluminum matrix. Attribution of this interaction to an aluminum-oxide interfacial layer should be supported by appropriate compositional or phase analysis.

Mahan H.M. and co-authors [14] similarly established that the alloy structure consists of numerous interdendritic matrix composites and fine precipitates dispersed throughout the volume.

Analysis of the composite surface microstructure confirms the intense interaction between the modified calcite particles, coated with an aluminum oxide microfilm, and the aluminum alloy, resulting in a high degree of material homogeneity. In the microstructure of the aluminum matrix composite, the phase components of the constituent materials are clearly identifiable, further confirmed by spectral analysis of the composite's elemental composition.

Study [15] discusses the main factors influencing the behavior of concrete-based radiation-shielding materials, including their physical, chemical, and morphological characteristics, as well as their effects on shielding performance, identifying national, material-specific, and emerging research trends that may stimulate further investigation in this promising field.

Particular attention has also been directed toward developing cost-effective, safe, and environmentally sus-

tainable materials. The literature [16] provides a detailed analysis of shielding systems based on ultra-high-performance concrete, presenting a comparative assessment of the trade-off between structural durability and radiation-shielding efficiency while highlighting practical limitations and existing research gaps.

Study [17] explores the fundamental nature of radiation and its interaction with matter, emphasizing X-rays and gamma rays and revealing their unique properties and wide range of applications in scientific and medical domains, providing a foundational understanding for navigating the complex nature of radiation across scientific, medical, and everyday contexts.

Nanomaterials in various forms have also attracted considerable attention in radiation protection due to their outstanding physicochemical properties. Study [18] reviews the fundamentals of radiation and radiation protection, lead-based shielding materials, nanomaterials, synthesis and characterization methods, polymers, and current research efforts aimed at developing nanocomposites as effective radiation attenuators and protective materials.

The findings reported in Study [19] demonstrate that magnetite-based high-strength concrete offers a more sustainable, cost-effective, and ethically justified alternative for radiation shielding in healthcare, nuclear, and industrial applications. The scientific significance of this work lies in its development of a transferable methodological framework that integrates life cycle assessment, life cycle cost analysis, and performance-normalized indicators.

The use of advanced, efficient, and environmentally sustainable materials is recognized as a key driver of global environmental, social, and economic transformation [20].

A comprehensive review of the literature [21–23] reveals very few studies specifically dedicated to aluminum matrix composites reinforced with calcium carbonate as

a filler for radiation-shielding applications. This gap has motivated the present study to investigate the radiation-shielding properties of aluminum matrix composites with calcite filler.

The measured dose rates for the aluminum-matrix composites containing calcite filler are presented in **Table 2**.

Table 2: Radiation shielding properties of aluminum matrix composite material in the form of 10 mm thick plates.

Sample No.	Number of Measurements					Average Value, $\mu\text{Sv/h}$
	1	2	3	4	5	
Background dose rate ($\mu\text{Sv/h}$)	0.24	0.28	0.26	0.24	0.26	0.25
Source ^{60}Co						
Source dose rate	3.91	2.82	2.70	2.62	2.68	2.94
Sample 1	2.40	2.33	2.12	2.18	1.33	2.07
Sample 2	2.14	2.07	2.10	1.84	1.88	2.01
Sample 3	2.12	2.19	2.10	1.82	1.69	1.98
Sample 4	2.25	2.22	2.20	1.66	1.38	1.94
Source ^{133}Ba						
Source dose rate	0.79	0.82	0.81	0.83	0.78	0.80
Sample 1	0.44	0.42	0.43	0.41	0.45	0.43
Sample 2	0.39	0.41	0.40	0.42	0.38	0.40
Sample 3	0.38	0.40	0.39	0.41	0.37	0.39
Sample 4	0.34	0.36	0.35	0.37	0.33	0.35
X-ray tube source: 70 kV, 200 mA, 0,080 s, 16 mAs						
Source dose rate	1485.0	1495.0	1490.0	1492.0	1488.0	1490.0
Sample 1	628.0	688.0	700.0	680.0	694.0	678.0
Sample 2	612.0	663.0	645.0	614.0	617.0	630.2
Sample 3	417.0	410.0	415.0	408.0	406.0	411.2
Sample 4	135.0	134.0	133.0	136.0	135.0	135.0

The analysis showed that Sample No. 4, with a thickness of 10 mm, provides the highest attenuation of gamma and X-ray radiation, reducing the dose rate by 1.5-fold (for ^{60}Co), 2.3-fold (for ^{133}Ba), and 11-fold (for X-ray radiation)—confirming the material’s radiation-protection properties.

5. Conclusions

Modification of the calcite surface enhances the interfacial interaction between the filler and the aluminum matrix, improving adhesion and the efficiency of mechanical

stress transfer. The uniform distribution of particles ensures formation of a dense, homogeneous structure, which positively affects the material’s radiation-protection properties.

The study results confirm the potential of the developed metal matrix composite based on modified natural calcite. The selected phase composition and uniform microstructure contribute to improved operational and protective characteristics.

The practical significance of this work lies in the possibility of using natural calcite from Tajikistan as an accessible and environmentally safe raw material for pro-

ducing special-purpose construction materials. The developed composite can be regarded as an economically efficient alternative to traditional radiation-protection materials.

List of Abbreviations

Al	Aluminum
AlCl ₃ ·6H ₂ O	Aluminum Chloride Hexahydrate
Ba	Barium
Ca	Calcium
CaCl ₂ ·2H ₂ O	Calcium Chloride Dihydrate
CaCO ₃	Calcium Carbonate
CO ₂	Carbon Dioxide
DTA	Differential Thermal Analysis
GOST	Gosudarstvennyy Standart
JEOL	Japan Electron Optics Laboratory
MeV	Megaelectronvolt
mm	Millimeter
MPa	Megapascal
TG	Thermogravimetric Curve/Analysis
TiO ₂	Titanium Dioxide
wt. %	Weight Percent
ZnO	Zinc Oxide
μSv/h	Microsievert per hour

Author Contributions

Conceptualization, methodology, and writing—original draft preparation: K.M.N.; Investigation, data curation, and visualization: D.I.M.; Investigation, formal analysis, and validation: M.M.I.; Supervision, project administration, and writing—review and editing: I.M. All authors have read and agreed to the published version of the manuscript.

Availability of Data and Materials

Data supporting the results of this study are available from the corresponding author upon reasonable request.

Conflicts of Interest

The authors declare no conflicts of interest.

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AI Declaration

During preparation of this manuscript, the authors used ChatGPT (OpenAI) to help improve the clarity and language of certain sentences. The AI tool was used solely for language editing, and the authors take full responsibility for the accuracy, originality, integrity, and scientific content of the manuscript.

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