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

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Abstract

The possibility of using calcite-shell limestone, widespread in the territory of Tajikistan, as a filler for an aluminum matrix in the synthesis of a metal matrix composite has been studied. The use of aluminum as a matrix allows for the production of the composite at a relatively low temperature (700 °C), avoiding the intense decomposition of calcium carbonate, which begins at 800-900 °C. The structural and phase changes of the material during the activation and modification of calcite have been studied. It has been shown that the 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. It has been established that a composite material consisting of 20 wt.% aluminum and 80 wt.% calcite with a thickness of 10 mm provides the highest attenuation of gamma- and X-ray radiation. The reduction in the dose rate from an open source from 1490 to 135 μSv/h (approximately 11 times) indicates the pronounced radiation protection properties of the developed material.

Keywords

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

Introduction

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

Thanks to calcite, durable and long-lasting binding materials are produced, capable of maintaining their operational properties both indoors and outdoors for many years [3, 4].

The subsoil of Tajikistan is characterized by significant reserves of natural minerals such as barite, calcite, bentonite, and several others, which possess promising physicochemical properties for use in radiation-shielding composite materials [5].

In this regard, scientific research aimed at the search, development, and scientific justification of technologies for producing environmentally safe building materials 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 the enhancement of the mechanical properties of metals. The introduction of calcium carbonate particles into the metal matrix exerts a strengthening effect, contributing to an increase in hardness, yield strength, and tensile strength.

One of the main problems in creating such composites remains porosity, as well as 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 considered to be 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, the intensive decomposition of calcium carbonate begins, releasing carbon dioxide. The released gas can cause the formation of pores and internal defects in the metal matrix. Therefore, modern research pays special attention to the development of protective coatings for calcium carbonate particles, as well as the application of low-temperature sintering methods.

The use of aluminum as a matrix in the creation of composite materials is due to the relative stability of the metal's physical and mechanical properties over a wide range of temperatures. This makes it possible to obtain composite materials with specified operational characteristics while optimizing production technological parameters and reducing the environmental impact.

The aim of the study is to investigate the features of synthesizing a metal-matrix composite based on natural calcium carbonate, as well as 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 mineral and an inexpensive natural material in Tajikistan. Its use as a filler in the development of new radiation-shielding metal-matrix composites is promising from both economic and environmental perspectives.

Materials and methods

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 point sample consisted of three mini-samples weighing 200-300 g, taken from the upper and middle layers. Then, the mini-samples were 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 activation and modification of natural calcium carbonate, the following substances were used:

-$CaCl_2 \cdot 2H_2O$ (GOST 450-77), manufacturer – "PrimeChemicalsGroup," Russia;

-$AlCl_3 \cdot 6H_2O$ (GOST 3759-75), manufacturer – LLC "Component-Reactive," Russia.

Sample preparation:

At the first stage, aqueous solutions of $CaCl_2 \cdot 2H_2O$ (0.7 M) and $AlCl_3 \cdot 6H_2O$ (0.1 M) were prepared.

To activate 10 g of calcite, it was mixed in 100 ml of water, after which 20 ml of a $CaCl_2 \cdot 2H_2O$ (0.7 M) solution was added to the suspension, and mixing continued for 30 minutes. Then the material was filtered and dried at a temperature of 80 °C.

For modification, 10 g of activated calcite was mixed in 100 ml of water with the addition of 30 ml of $AlCl_3 \cdot 6H_2O$ (0.1 M) solution. The resulting mixture was stirred with a mechanical stirrer for 30 minutes, after which the material was washed and dried at a temperature of 120-160 °C.

Methods

Synthesis of a composite:

Metal matrix composite material in the form of four samples with the following composition:

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

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

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

Role of components in the composite:

-calcite (filler) – contributes to the reduction of gamma and X-ray radiation intensity;

-$CaCl_2 \cdot 2H_2O$ (activator) – activates the surface of calcite;

-$AlCl_3 \cdot 6H_2O$ (modifier) – modifies the surface of calcite;

-metallic aluminum (matrix) – imparts lightness, plasticity, non-toxicity, and corrosion resistance to the composite material.

X-ray Phase Analysis:

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

During the study, differential thermal analysis was used to investigate the thermal transformations of the initial mineral raw material. Thermal studies were conducted using the Labsys Evo-C1600 derivatograph manufactured by Setaram Instrumentation (France). This method allows determining 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 studied using a JEOL JSM-IT200 scanning electron microscope (Japan).

The tests of the radiation protection properties of the composites were conducted using gamma radiation sources:

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

-^{133}Ba (E = 0.35 MeV);

-dosimeter ESMFH40G-L10 THERMO (Germany),

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calibration – February 2026.

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

The determination of the attenuation level of the X-ray dose power was conducted on the SF-80 X-ray machine (Japan) using the DKS AT 1121 dosimeter (Republic of Belarus) with equipment that excludes the influence of scattered radiation.

Testing conditions:

-distance to the source – 1 m;

-positioning – lateral;

-X-ray tube parameters: 70 kV, 200 mA, 0.080 s, 16 mAs.

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%. For this phase, the most intense diffraction peaks are characteristic, forming the main part of the X-ray diffraction pattern (Fig. 1), which indicates its predominant content in the material's structure.

#	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

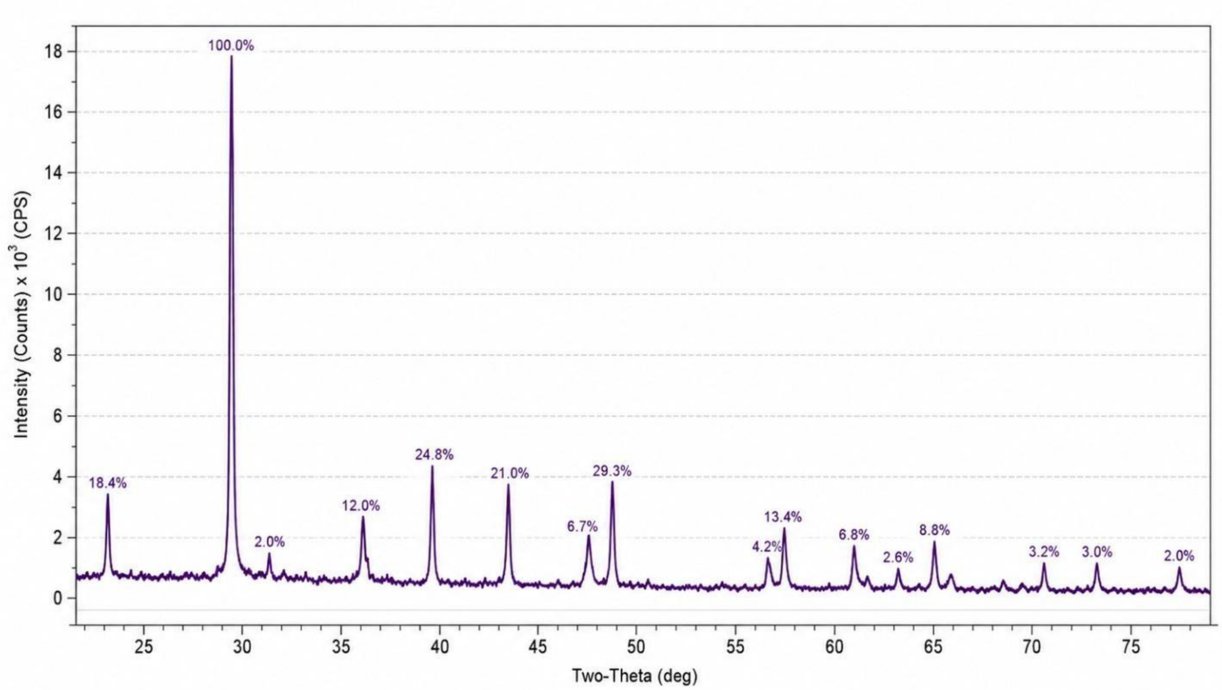

Figure 1: X-ray phase analysis of calcite-limestone.

As a secondary phase in the sample, zinc oxide (ZnO) has been identified, with a content of approximately 6.3% by mass. The presence of this phase is confirmed by characteristic diffraction peaks of medium intensity.

Moreover, in a small amount (about 2.4%), the rutile (TiO_2) phase was detected in the sample, the presence of which is confirmed by weakly expressed peaks on the diffractogram.

The distribution and position of the diffraction maxima on the X-ray diffraction pattern are in good agreement with the reference data for the specified crystalline phases. The obtained 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 the shell limestone shows its proximity to the composition of calcite, which is characterized by a content of about 56% CaO and 44.0% CO_2.

Figure 2 presents the thermal curves of the calcite-

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shell, reflecting the dependence of the sample mass change (TG curve) and thermal effects (DTA curve) on the heating temperature.

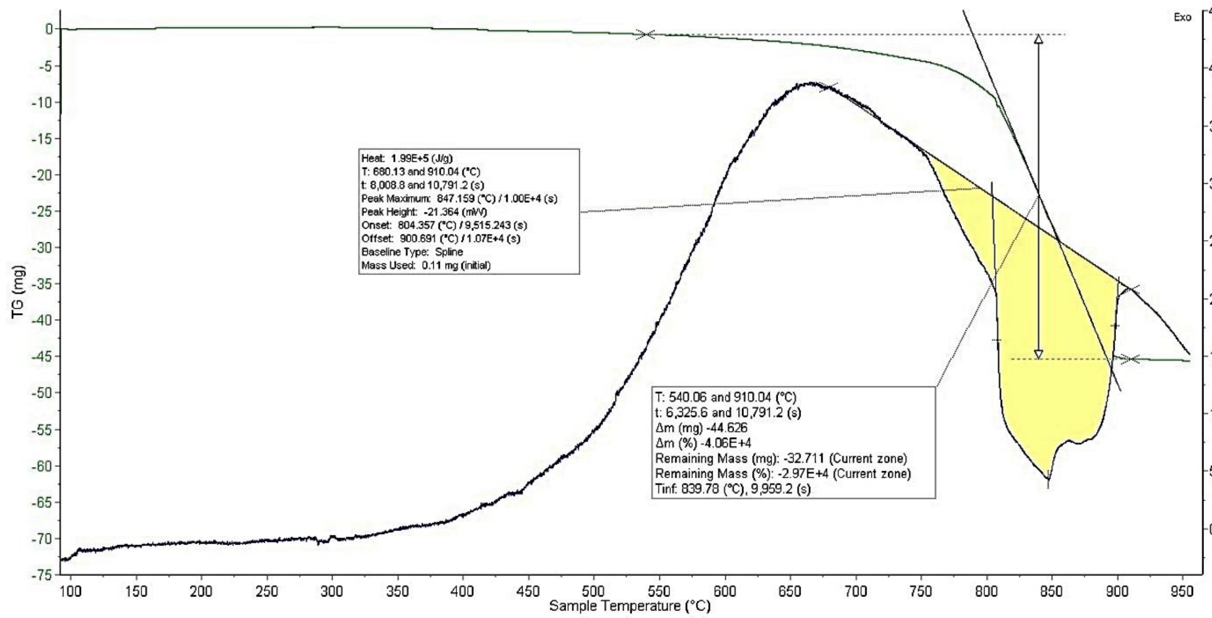

Figure 2: Thermal curves of calcite-limestone.

The analysis of thermograms shows the presence of a pronounced endothermic effect at a temperature of about 847.2 °C, accompanied by a mass loss of the sample by 44.626 mg. This effect is associated with the thermal decomposition of the carbonate phase and the re-lease of carbon dioxide. The thermal effect of the process is estimated at around 1.99×10^5 J/g, indicating the occurrence of intense thermochemical transformations in the structure of the calcite raw material.

The obtained results confirm the data of 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: $CaCO_3 \rightarrow CaO + CO_2\uparrow$.

The process occurs within a temperature range of 800-900 °C. The obtained results justify the choice of aluminum as the metal matrix. Due to the melting point of aluminum (660 °C), calcium carbonate is preserved in the composite as a filler without undergoing thermal decomposition.

The reason for usage of aluminum metal matrix composites is due to their low density and high-performance properties [9].

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

The modification of pre-activated calcite was carried out by the method of adsorptive fixation of ions on the particle surface using an aqueous solution of aluminum chloride.

The modification process is carried out due to the electrostatic interaction between the charged active sites of the particles and the ions of the modifying agent, as well as due to the ion-dipole interaction with the oxygen atoms of the hydroxyl groups present on the surface of the activated calcite particles. It should be noted that the high defectiveness of the calcite particle surfaces and the presence of a developed system of active centers create favorable conditions for the adsorptive fixation of modifying components (Fig.3).

Most likely, the nature of calcite particles exhibits a defective form with a significant degree of aggregation edge and roughness. The external surface of this order can be modified and described in a manner analogous to the final composition with a matrix to ensure greater interweaving between them.

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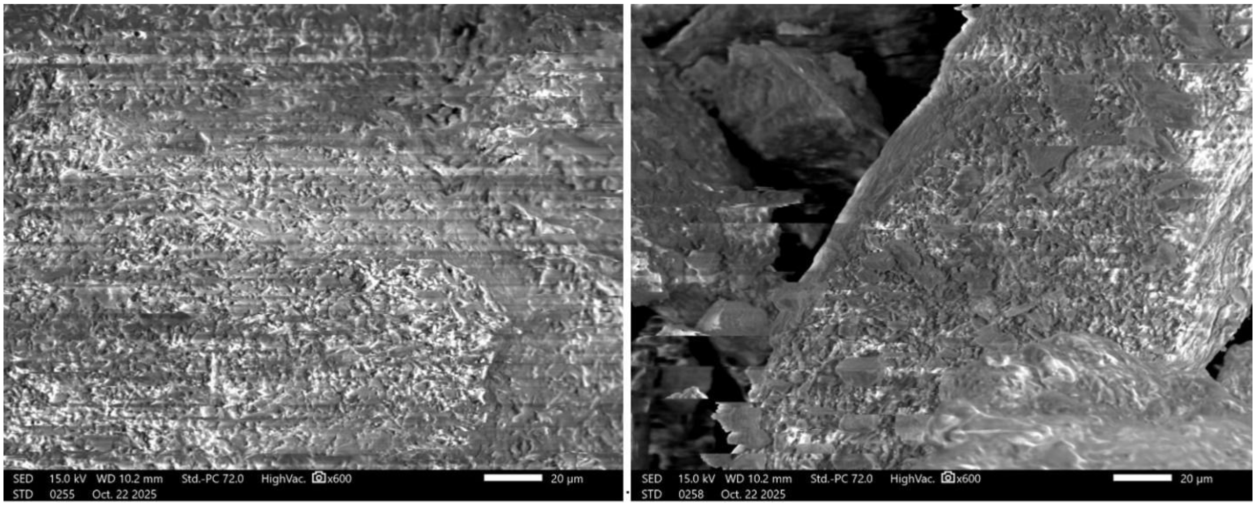

a) b)

Figure 3: Microstructure of calcite: a – unmodified; b – modified.

On the fixed external plane of calcite particles before modification and subsequently modified, it is possible to distinguish modified calcite from unmodified calcite by the appearance of a metallic luster due to the adsorbed layer of aluminum on its external plane.

Surface modification leads to an increase in the number of functional groups and the formation of a more developed interfacial layer, which contributes to improved interaction between the mineral filler and the metal matrix (Fig. 4).

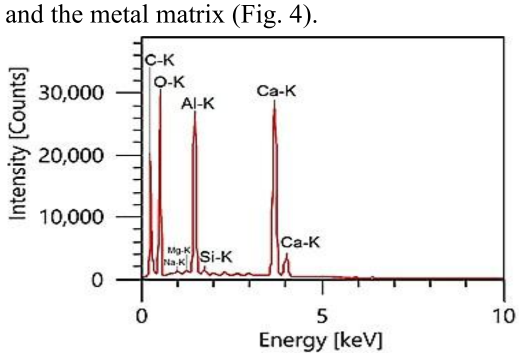

Figure 4: Spectra of modified calcite.

X-ray fluorescence analysis of modified calcite shows that the carbon content is 11.21 wt.%, oxygen 47.91 wt.%, calcium 39.41 wt.%, and impurities Al, Si, Na, Mg are the rest.

Discussion

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

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

The obtained modified calcite was used as a dispersive filler in the production of a metal-matrix composite. One of the key stages in the technology for producing metal matrix composite materials is the process of uniformly distributing filler particles in the metal matrix, as this factor largely determines the density, structural uniformity, mechanical properties, and functional characteristics of the material. When forming the aluminum matrix composite, special attention was paid to ensuring the uniform distribution of modified calcite particles in the aluminum melt. The technological process for obtaining the composite involved melting aluminum at a temperature of around 700 °C, followed by the introduction of modified calcite filler with intensive mechanical stirring until a homogeneous composite mixture was obtained (Fig. 5).

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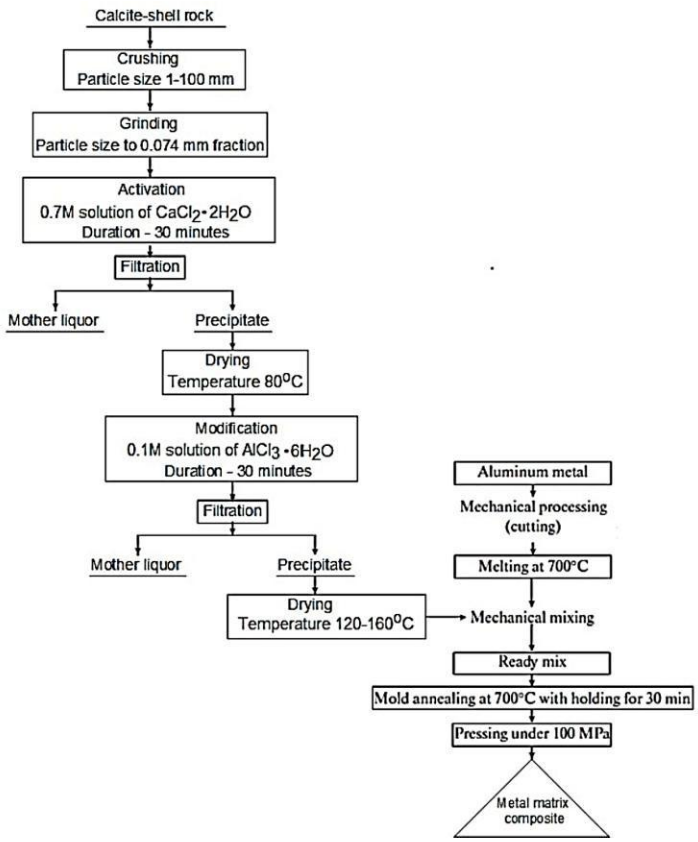

Figure 5: Technological scheme for obtaining a metal-matrix composite material based on calcite shell rock.

After obtaining the composite melt, the material was placed in a mold and subjected to thermal treatment at a temperature of 700 °C for 30 minutes. Next, the samples were subjected to pressing under a pressure of 100 MPa. As a result, metal-matrix composite materials in the form of 10 mm thick plates were obtained, intended for subsequent studies, including their radiation protection properties.

Earlier, in the work of Varun Singhal et al. (2025), the main focus was on the study of aluminum-based metal matrix composites. The authors analyzed their properties, the influence of aging processes, and the possibilities of predictive modeling, which allowed for an expanded understanding of the prospects for using these composites and their role in modern materials science [10].

MS Ayar et al. (2021) investigated the current state of development of aluminum metal matrix composites, emphasizing their ability to ensure material and energy resource savings. The paper discusses key technological issues related to the uniform distribution of the reinforcing phase and the high cost of processing, while the authors note significant prospects for further development and implementation of these composites in engineering practice [11].

In the study by Sayed Ali Ahmad Alem et al. (2025), metal matrix composites reinforced with metallic particles are examined. The authors note that such reinforcing materials possess a unique combination of high physical and mechanical properties, ensuring enhanced operational characteristics of the composites. Compared to traditional ceramic reinforcing materials, metallic particles demonstrate higher compatibility with metallic matrices. Particular attention in this work is given to the study of interaction mechanisms at the interface between the matrix and the reinforcing phase, as well as the analysis of technological processes necessary for obtaining highly efficient metal matrix composites [12].

In study [13], aluminum was employed as the matrix material for the fabrication of 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, the formation of a stable interfacial region is expected, enabling the synthesis of the composite

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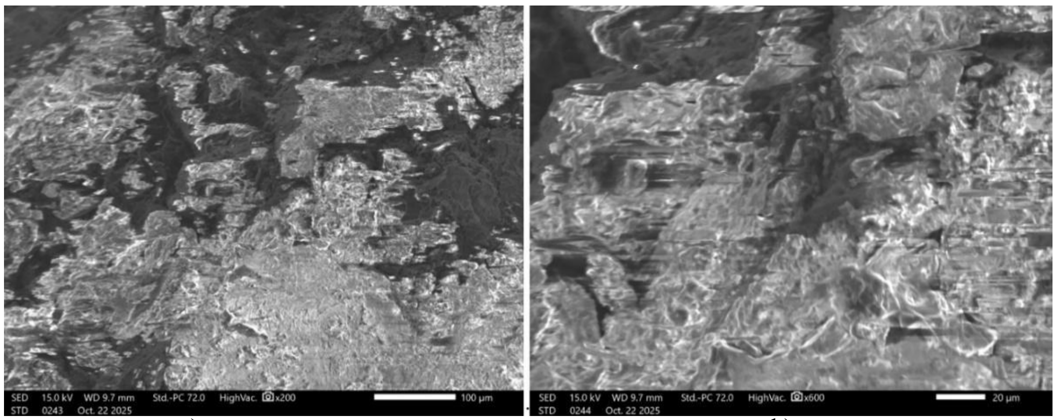
at a relatively low temperature of 770 °C without decomposition or loss of the calcite filler.The analysis of the microstructure of the aluminum matrix composites we obtained with calcite fillers of various dispersities showed that the manufactured samples exhibit a uniform distribution of aluminum throughout the entire volume of the composite (Fig. 6).



a) b)

Figure 6: Microstructure of the aluminum matrix composite: a – at x200 magnification; b – at x600 magnification.

At high magnification, it can be seen that the particles are located at a small distance from each other, and the gaps between them are filled with aluminum. This indicates a good interaction between the calcite particles and the aluminum alloy during the composite formation process. Such interaction is due to the presence of a microfilm of aluminum oxide, which is fixed on the surface of the particles during the modification process. The microfilm contributed to the strong adhesion of calcite to the aluminum matrix, ensuring the complete coating of the filler particles with the aluminum alloy.

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

The study of the microstructure of the composite surface confirms the intense interaction of modified calcite particles, coated with an aluminum oxide microfilm, with the aluminum alloy, ensuring a high degree of material homogeneity. In the microstructure of the aluminum matrix composite, the phase components of the used materials are distinctly identifiable, which is further confirmed by the results of the spectral analysis of the elemental composition of the composite phases.

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 the resulting effects on their shielding performance. National, material-specific, and emerging research trends are identified, which may stimulate further investigations in this promising field.

It is also important to note that particular attention has been focused on the development of cost-effective, safe, and environmentally sustainable materials. The literature [16] provides a detailed analysis of shielding systems based on ultra-high-performance concrete and presents a comparative assessment of the trade-off between structural durability and radiation shielding efficiency, highlighting practical limitations and existing research gaps.

Furthermore, study [17] explores the fundamental nature of radiation and its interaction with matter. It emphasizes the field of X-rays and gamma rays, revealing their unique properties and wide range of applications in scientific and medical domains. This provides a foundational understanding for navigating the complex nature of radiation in various scientific, medical, and everyday contexts.

Nanomaterials in various forms have also attracted significant attention in the field of 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.

Study [19] demonstrates 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. Moreover, the scientific significance of this work lies in the development of a transferable methodological framework that integrates life cycle assessment, life cycle

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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 that there are very few studies specifically dedicated to aluminum matrix composites reinforced with calcium carbonate as a filler for radiation shielding applications. This gap in the literature has motivated the present study to investigate the radiation-shielding properties of aluminum matrix composites with calcite filler.

The results of the studies on the radiation-shielding properties of the aluminum matrix composite material with calcite filler are presented in Table 1.

Table 1. Radiation shielding properties of aluminum matrix composite material in the form of 10 mm thick plates

Sample No.	**Number of measurements**					**Average value, μSv/h**
	1	**2**	**3**	**4**	**5**	
Background dose rate (μSv/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 кv, 200 мA, 0,080 s, 16 мAs						
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. Depending on the radiation sources, it reduces the dose rate by 1.5 times (for ^{60}Co), 2.3 times (for ^{133}Ba), and 11 times (for X-ray radiation), which indicates the radiation protection properties of the material.

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 the formation of a dense and homogeneous structure, which positively affects the radiation protection properties of the material.

The results of the study confirm the promise of the

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developed metal matrix composite based on modified natural calcite. It has been established that the optimized phase composition and uniform microstructure contribute to the improvement of operational and protective characteristics.

The practical significance of the work lies in the possibility of using natural calcite from Tajikistan as an accessible and environmentally safe raw material for the creation of special-purpose construction materials. The developed composite can be considered an economically efficient alternative to traditional radiation protection materials.

List of Abbreviations

Al – Aluminum
$CaCl_2 \cdot 2H_2O$ – Calcium Chloride Dihydrate
$AlCl_3 \cdot 6H_2O$ – Aluminum Chloride Hexahydrate
DTA – Differential Thermal Analysis
TG – Thermogravimetric curve/analysis
TiO_2 – Titanium Dioxide
ZnO – Zinc Oxide
MPa – Megapascal
wt.% – Weight Percent
mm – Millimeter
µSv/h – Microsievert per hour
MeV – Megaelectronvolt

Ba	Barium
Ca	Calcium
$CaCO_3$	Calcium carbonate
CO_2	Carbon dioxide
JEOL	Japan Electron Optics Laboratory

Authors' Contributions

Conceptualization, methodology, writing – original draft preparation: Nazarov Kh.M.; Investigation, data curation, visualization: Mirzoev D.I.; Investigation, formal analysis, validation: Ismatdinov M.M.; Supervision, project administration, writing – review and editing: Mirsaidzoda I. All authors have reviewed and approved the final version of the manuscript

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Conflicts of Interest

The authors state that there are no potential conflicts of interest.

Availability of data and materials

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

Funding

No external funding was received for this research. This work was carried out as part of the research plan Chemical, Biological, Radiological and Nuclear Safety and Security Agency of the National Academy of Sciences of Tajikistan on the topic: "Features of the extraction of uranium concentrate and other valuable components from ores and waste of uranium production in Tajikistan" (Years of implementation: 2025-2029). State Registration No. 0125 TJ 1658.

Acknowledgments

The authors express their gratitude to the staff of the Technical Services Laboratory for Chemical, Biological, Radiological and Nuclear Safety and Security Agency of the National Academy of Sciences of Tajikistan for their assistance in organizing and conducting the research.

The authors are also grateful to the reviewers for their valuable comments and recommendations, which contributed to the improvement of the article's quality.

AI-declaration

During the preparation of this manuscript, the authors used ChatGPT (OpenAI) to assist in improving the clarity and language of certain sentences. The AI tool was used solely for language editing purposes, and the authors take full responsibility for the content of the manuscript.

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