

## PREPARATION OF CALCIUM TITANATE PEROVSKITE COMPOUND, OPTICAL AND STRUCTURAL PROPERTIES

✉ **Khujamkul T. Davranov**<sup>a</sup>, ✉ **Muradulla T. Normuradov**<sup>a</sup>, ✉ **Muzaffar A. Davlatov**<sup>b</sup>,  
✉ **Kuvondiq T. Dovranov**<sup>a\*</sup>, **T.U. Toshev**<sup>b</sup>, **N.A. Kurbonov**<sup>b</sup>

<sup>a</sup>Karshi State University, Karshi, 180100, Uzbekistan

<sup>b</sup>Karshi Engineering and Economic Institute, Karshi, 180100, Uzbekistan

\*Corresponding Author e-mail: [quvondiqdavronm@gmail.com](mailto:quvondiqdavronm@gmail.com)

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In this work, we have successfully fabricated a calcium titanate perovskite compound. The resulting CaTiO<sub>3</sub> compound was studied by preparing samples by compacting it in a powder state and using a Pousson device. The distance between the planes  $d_{hkl}$ , Miller indices (hkl), degree of crystallinity and amorphism, structure and lattice parameters of the calcium titanate perovskite compound were determined using an X-ray diffractometer. Also, according to the results of FT-IR analysis, the formation of CaTiO<sub>3</sub> perovskite is confirmed as a result of the study of molecular vibrations. The main broad peaks are observed in the range of 680–400 cm<sup>-1</sup>, the absorption band at the wave number of 543,93 cm<sup>-1</sup> corresponds to the specific stretching vibrations of Ti-O bonds and indicates the formation of the CaTiO<sub>3</sub> perovskite type structure implies. Based on the results of these measurements, it will be possible to use semiconductor compounds in the future to create nanofilms by magnetron sputtering.

**Keywords:** X-ray phase analysis; Miller indices; Interplanar distance; Calcium titanate; Crystalline and amorphous phases; FT-IR  
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### INTRODUCTION

Along with the rapid development of science and technology, the demand for energy is increasing day by day. The world is rapidly depleting non-renewable energy sources such as hydrocarbon fuels. Therefore, humanity needs to find alternative ways to solve these pressing problems. One of the most effective ways to combat global climate change is concrete measures such as replacing fossil fuels with new energy-efficient materials and renewable energy sources [1]. Based on this, the developed countries of the world are increasing their investments in this field every year in order to achieve sustainable development of new technologies and green energy [2]. Among renewable energy sources, photovoltaic generators are the most promising way to replace fossil fuels. Photovoltaic generators are based on the properties and characteristics of semiconductor materials, and in recent years have been derived from polycrystalline and monocrystalline silicon compounds. Obtaining high-quality thin coatings of metals, alloys, and semiconductors is one of the most important tasks in the production technology of integrated circuit elements, various temperature and pressure sensors, and micro and nanoelectronics in general. In particular, semiconductor nanofilms with a narrow area are needed to create thermal sensors. Metal silicides are one such semiconductor. Therefore, obtaining thin-layered and thin-sized films from these semiconductors is an urgent task [3].

Many different methods are used to obtain thin film coatings. The most popular methods of vacuum deposition of coatings on various types of surfaces are thermal evaporation, electron beam evaporation and various types of ion-plasma spraying. In this case, only the methods of applying thin films are based on the sputtering of the material with ions of heavy gases. These include ion-beam and magnetron methods of deposition of thin-film structures, which have been intensively developed recently [4]. They have a number of advantages: new materials, the ability to obtain oxides, good film adhesion and physicochemical properties. Especially important in the production of microelectronics and solar energy elements, it is necessary to use multi-structured coatings such as metal-semiconductor-dielectric, etc., which requires the use of various vacuum plasma equipment [5].

In recent times, it is known to everyone that it is almost impossible to imagine electronics without semiconductor heterostructures. Such structures are widely used to create light-emitting diodes, short-wave photodetectors, semiconductor lasers, solar cells, and other products of modern optoelectronics. An important factor in the widespread use of such devices is low cost and resistance to high temperature conditions. Calcium titanate is a promising material for creating various optoelectronic devices based on it. Calcium titanate is a combination of barium and titanium oxides. Crystal modification of calcium titanate with perovskite structure is ferroelectric with photorefractive and piezoelectric effect. Calcium titanate is characterized by high values of dielectric conductivity, on the basis of which several types of ferroelectric ceramics have been developed, which are used to create capacitors, piezoelectric sensors and posistors [6].

In this work, we study the structural properties and optical spectra of CaTiO<sub>3</sub> through the absorption properties of X-rays and infrared spectrum of light, physicochemical properties.

## PREPARATION OF SAMPLES

The starting material used for synthesizing  $\text{CaTiO}_3$  was analytical reagent grade, initially mixing calcium (96%) powder to titanium oxide ( $\text{TiO}_2$ ) powder in a 1:1 ratio at room temperature using a digital mechanical stirrer for two hours. This mixture was stepwise calcined by heating in a muffle furnace at a rate of  $20\text{ }^\circ\text{C}/\text{min}$  increasing the temperature to the next higher temperature until the terminal temperature of the reaction mixture was reached. The resulting powder was thermally heated in a muffle furnace at  $800^\circ\text{C}$  for 1 hour, then heated to  $1100^\circ\text{C}$  for another 1 hour, and then naturally cooled to room temperature by heating to  $1300^\circ\text{C}$  for half an hour. The resulting  $\text{CaTiO}_3$  compound was studied by preparing samples by compacting it in a powder state and using a Pousson device.

## METHODS

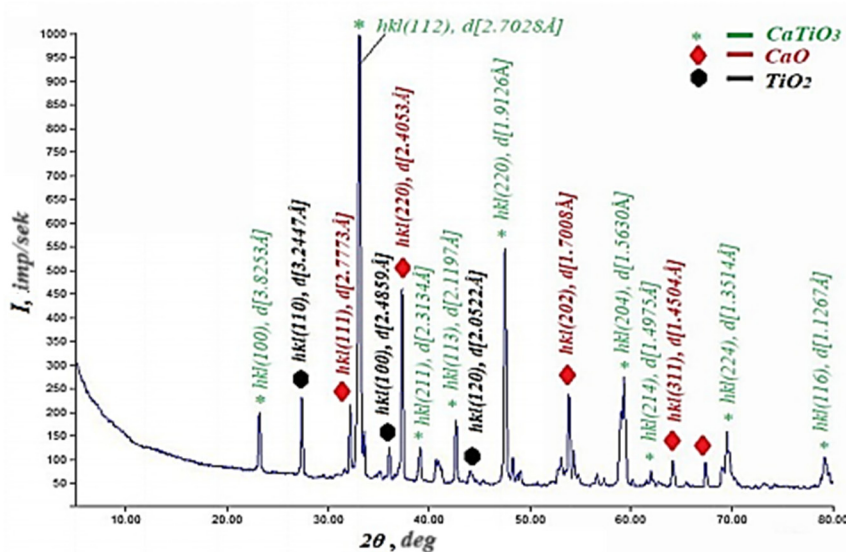
X-ray diffractometers and infrared spectrophotometers were used to determine the qualitative composition of the sample, the crystal structure of the substance, as well as the unit cell parameters (full profile analysis - Rietveld method), the crystal size of the polycrystalline sample (coherent scattering region). X-ray diffraction patterns were obtained using a XDR-6100 (Shumadzu) diffractometer with a Cu-K $\alpha$  ( $\beta$ - filter, Ni, current and voltage mode 30 mA, 40 kV) as a radiation source. Measurements were made with a step of 0.05 degrees, a constant detector rotation speed of 4 deg/min ( $\omega/2\theta$ -link) and the scanning angle varied from  $10^\circ$  to  $100^\circ$  at  $2\theta$ . The X-ray power was 2 kW. The results were analyzed using the database [7]. The penetration depth of Cu-K $\alpha$  radiation is about 1 mm (980  $\mu\text{m}$ ) for light elements (carbon), several microns for heavy elements (Ag, W). For most inorganic substances, Cu-K $\alpha$ - is tens of microns ( $\mu\text{m}$ ) for simple compounds.

Elemental analysis was performed using Fourier-transform infrared (FTIR) spectroscopy. Absorption and transmission spectra of the samples were obtained using the "Happ-Hanzel" method on an IRTracer-100 spectrophotometer. In order to reduce the effect of water vapor ( $\text{H}_2\text{O}$ ) and carbon dioxide ( $\text{CO}_2$ ) molecules in the sample in the range of  $400\div 4000\text{ cm}^{-1}$ , several of the following corrections were performed: addition, smoothing, main zero correction, normalization and ATR correction.

## RESULTS AND THEIR DISCUSSION

### X-ray diffraction analysis

Figure 1 shows the spectral relationships of the  $\text{CaTiO}_3$  compound obtained by the powder diffractometer method. In addition, the Miller indices are given, as well as the distance between planes  $d_{hkl}$  for these samples. We used the Rietveld method to refine the structure from X-ray powder data [7]. The principle of the method is to use independent intensity measurements at each point of the diffraction pattern, to describe the line profile using analytical functions, instead of using the integral reflection intensity. Feature parameters, including structural, device, and other characteristics, are refined using a nonlinear least-squares method. Using this refinement method, we determined the distance between the planes  $d_{hkl}$  and the Miller index (hkl). As mentioned above, powder X-ray diffraction allows for quantitative elemental analysis.



**Figure 1.** Spectral dependence  $\text{CaTiO}_3$ , is obtained by the method of powder diffractometer. Miller indices are given

The degree of crystallinity and amorphism was evaluated for the samples measured by X-ray diffraction analysis using Search and Match software [8]. For calcium titanate, the amorphous phase is 71.35% and the crystalline phase is 28.65%, respectively. In addition, the phase composition (Weight %) and elemental composition of the  $\text{CaTiO}_3$  sample was determined using the "Profex" program. The phase composition of the sample prepared for magnetron spraying corresponds to 90.7% perovskite ( $\text{CaTiO}_3$ ), 3.8% titanium, 5.5% calcium. Our elemental analysis of the samples using

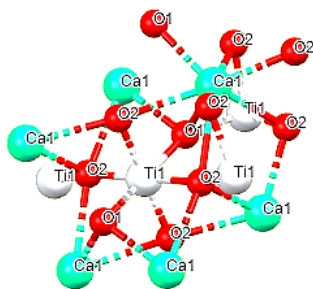
Search and Match software shows that the samples have the following composition (in weight percent): 35.7% Ti, 32.2% Ca, and 32.0% O.

As is known from the literature data [9], Miller indices are applicable in all synagogues. As the Miller index increases, the interplanar spacing decreases [3].

**Table 1.** X-ray analysis of CaTiO<sub>3</sub> perovskite: Miller index, 2theta, intensities, distance between planes, FWHM

No.	hkl	2theta [°]	d [Å]	I/I <sub>0</sub> (peak height)	Counts (peak area)	FWHM
1	100	23.22	3.8253	120.92	396.92	0.2000
2	110	27.42	3.2447	159.16	417.97	0.1600
3	111	32.20	2.7773	147.20	386.55	0.1600
4	112	33.12	2.7028	1000.00	3939.12	0.2400
5	220	37.36	2.4053	401.92	1055.47	0.1600
6	220	47.54	1.9126	512.76	2019.83	0.2400
7	202	53.86	1.7008	162.85	320.75	0.1200
8	204	59.04	1.5630	157.46	1033.76	0.4000
9	214	62.12	1.4975	181.27	952.06	0.3200
10	224	69.50	1.3514	110.16	361.61	0.2000

Figure 2 shows an X-ray image of a CaTiO<sub>3</sub> compound measured at room temperature. X-ray phase analysis at room temperature shows that peaks at different degrees 23.22, 27.42, 32.20, 33.12, 37.36, 47.54, 53.86, 59.04, 59.32, 69.50 are visible at several angles.



**Figure 2.** Crystal structure (orthorhombic structure sp. gr. Pbnm)

Processing by the full-profile method of X-ray diffraction data showed that the sample has an orthorhombic structure (sp. gr. Pbnm) with the following lattice parameters: a = 5.3064 Å, b = 5.3531 Å, c = 7.1760 Å and positions, coordinates of atoms in the unit cell (Table 2).

**Table 2.** Comparative analysis of lattice parameters with the literature

Lattice parameters	a/ Å	b/ Å	c/ Å
This work	5.3064	5.3531	7.1760
Literature [12]	5.380	5.443	7.640

Figure 2 shows the crystal structure CaTiO<sub>3</sub> of calcium titanate, the unit cell of the crystal lattice CaTiO<sub>3</sub> corresponds to the orthorhombic space group and consists of three chemical formulas: Ca, Ti, O.

### Infrared spectroscopy analysis

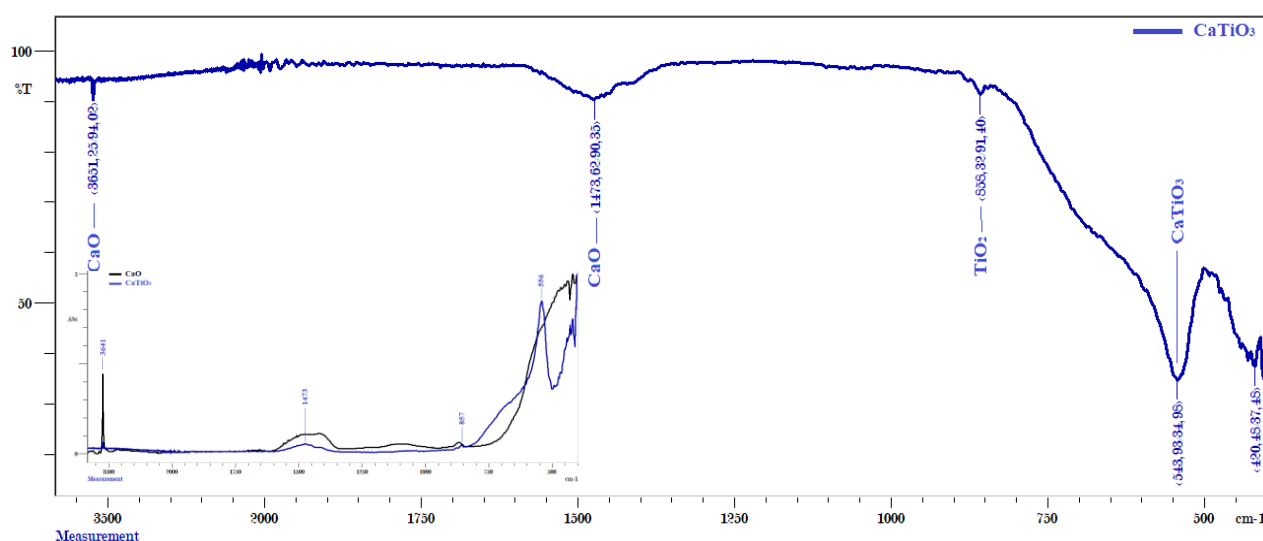
Powder absorption and transmission infrared (IR) spectra of perovskite CaTiO<sub>3</sub> annealed several times at different temperatures were investigated in the range of 400÷4000 cm<sup>-1</sup> at room temperature. The spectra vary depending on the composition, annealing temperature and structural state. Autocorrelation analysis was used to determine the variation of the average line width of groups of peaks in primary IR spectra [13-15]. Fourier transform infrared spectroscopy (FTIR) analysis of CaTiO<sub>3</sub> synthesized at high temperature (1300°C) was carried out and its spectrum is shown in Fig. 3. It can be seen from the transmission spectrum that there is a band at the wave number 3600 ÷ 3800 cm<sup>-1</sup>. It corresponds to the symmetric and asymmetric stretching vibration of weakly bound water interacting with the environment through hydrogen bonding and the O - H stretching modes corresponding to the stretching vibrations of hydrogen-bonded OH groups. As the molar ratio increased to 1:4, no peak was found indicating the absence of moisture and water molecules in this system [16]. Therefore, a molar ratio of 1:4 is the best condition for the anhydrous formation of CaTiO<sub>3</sub> perovskite powders.

The main broad peaks are observed in the range of 680÷400 cm<sup>-1</sup>, the absorption band at the wave number of 543.93 cm<sup>-1</sup> corresponds to the specific stretching vibrations of Ti-O bonds, which indicates the presence of TiO<sub>6</sub> octahedra and CaTiO<sub>3</sub> implies the formation of a perovskite-type structure [17]. The band at wave number 435.91 cm<sup>-1</sup> is characteristic of Ca-Ti-O bending vibrations of calcium titanate. It is due to a weaker, symmetric stretching band (C-O) in the range of 1480÷1380 cm<sup>-1</sup> [18]. The strongest peaks correspond to the wave number of 543.93 cm<sup>-1</sup>, 435.91 cm<sup>-1</sup>, 420.48 cm<sup>-1</sup>, 405.05 cm<sup>-1</sup>, characteristic vibrations of calcium titanate in the range of 910÷3600 cm<sup>-1</sup> at 1100 °C. its

absence indicates the formation of pure  $\text{CaTiO}_3$  [19]. This clearly shows that thermal annealing can be one of the well-established synthetic routes to prepare calcium titanate perovskite materials. Table 3 presents several correlations and wavenumber intensities in the absorption spectrum of a sample prepared using a stoichiometric mixture.

**Table 3.** Multiple correlations and intensity in the absorption spectrum of  $\text{CaTiO}_3$

no	Peak Intensity	Corr. Intensity	Base (H)	Base (L)	Area Corr.	Area	Area color
1	405.05	24.00	5.71	410.84	401.19	699.526	29.405
2	420.43	28.43	5.06	422.41	420.84	802.448	32.825
3	543.93	31.05	3.90	553.27	532.05	1395.688	50.320
4	858.32	23.13	23.68	857.93	857.63	6531.839	1185.06
5	1473.62	83.92	6.46	1455.11	1422.32	398.420	82.770



**Figure 3.** FTIR transmission and absorption spectrums of  $\text{CaTiO}_3$  prepared at  $1300^\circ\text{C}$

The results of X-ray and IR absorption measurements of the  $\text{CaTiO}_3$  perovskite compound show that it is one of the most versatile oxides of the perovskite family for applications in fields such as electronics and photovoltaics due to its structural structure and optical properties. Therefore, it is important to study the ways of forming semiconductor material.

## CONCLUSION

The qualitative composition of  $\text{CaTiO}_3$  perovskite compound formed by mechanical mixing and thermal heating, the amorphous and crystalline phase of the substance, and its structure were studied with the help of XDR-6100 type diffractometer. Using "Fullprof", "Profex" processing programs, unit cell parameters, Milner indices and interplanar distance of the sample were determined. It was observed that as the Miller index increased, the distance between the planes decreased. For calcium titanate, the amorphous phase is 71.35% and the crystalline phase is 28.65%, respectively. The phase composition of the sample corresponds to 90.7% perovskite ( $\text{CaTiO}_3$ ), 3.8% titanium, 5.5% calcium. Elemental analysis shows that the samples have the following composition (in weight percent): 35.7% – Ti, 32.2% – Ca and 32.0% – O. FT-IR analyzes of the  $\text{CaTiO}_3$  compound were conducted. The main broad peaks are observed in the range of  $680\div 400\text{ cm}^{-1}$ , the absorption band at the wave number of  $538.14\text{ cm}^{-1}$  corresponds to the specific stretching vibrations of Ti-O bonds and indicates the formation of the  $\text{CaTiO}_3$  perovskite type structure implies. The band at wave number  $435.91\text{ cm}^{-1}$  is characteristic of Ca-Ti-O bending vibrations of calcium titanate. The strongest peaks correspond to the wave numbers of  $543.93\text{ cm}^{-1}$ ,  $435.91\text{ cm}^{-1}$ ,  $420.48\text{ cm}^{-1}$ ,  $405.05\text{ cm}^{-1}$ . Absence of characteristic vibrations of calcium titanate in the range of  $910\div 3600\text{ cm}^{-1}$  at  $1100^\circ\text{C}$  indicates the formation of calcium titanate perovskite. Based on the results of these measurements, it will be possible to use semiconductor compounds in the future to create nanofilms by magnetron sputtering.

## ORCID

© Khujamkul Davranov, <https://orcid.org/0000-0002-2373-3026>; © Muradulla Normuradov, <https://orcid.org/0000-0003-1771-0853>  
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**ОТРИМАННЯ ПЕРОВСКІТНОЇ СПОЛУКИ ТИТАНАТУ КАЛЬЦІУ, ОПТИЧНІ ТА СТРУКТУРНІ ВЛАСТИВОСТІ**  
**Худжамкул Т. Давранов<sup>а</sup>, Мураддулла Т. Нормурадov<sup>а</sup>, Музаффар А. Давлатов<sup>б</sup>, Кувондик Т. Довранов<sup>а</sup>,**  
**Т.У. Тошев<sup>б</sup>, Н.А. Курбонов<sup>б</sup>**

<sup>а</sup>Каршинський державний університет, Карші, 180100, Узбекистан

<sup>б</sup>Каршинський інженерно-економічний інститут, Карші, 180100, Узбекистан

У цій роботі ми успішно виготовили перовскітну сполуку титанату кальцію. Отриману сполуку CaTiO<sub>3</sub> досліджували шляхом приготування зразків пресуванням у порошкоподібному стані та використанням приладу Пуссона. За допомогою рентгенівського дифрактометра визначали відстань між площинами dhkl, індекси Міллера (hkl), ступінь кристалічності та аморфізму, структуру та параметри решітки перовскітної сполуки титанату кальцію. Також за результатами FT-IR аналізу підтверджено утворення перовскіту CaTiO<sub>3</sub> в результаті дослідження молекулярних коливань. Основні широкі піки спостерігаються в діапазоні 680÷400 см<sup>-1</sup>, смуга поглинання на хвильовому числі 543,93 см<sup>-1</sup> відповідає питомим валентним коливанням зв'язків Ti-O і свідчить про утворення передбаченої перовскітної структури CaTiO<sub>3</sub>. За результатами цих вимірювань у майбутньому можна буде використовувати напівпровідникові сполуки для створення наноплівки методом магнетронного розпилення.

**Ключові слова:** рентгенофазовий аналіз; індекси Міллера; міжплощинна відстань; титанат кальцію; кристалічна та аморфна фази; FT-IR