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Study of the thermal conductivity of natural carbonates

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Abstract

The thermal conductivity of natural monoliths of calcite, dolomite marble, and limestone from various deposits was measured using the absolute stationary method of longitudinal heat flow in the temperature range of 50–300 K and the dynamic method in the range of 323–573 K. A majority of calcite marbles were inferior in thermal conductivity to dolomite marbles. At room temperature, the thermal conductivity coefficients of all studied samples were lower $k = 5$ W/(m K).

The obtained data were compared with the literature data. The diversity of experimental data from different authors on the thermal conductivity of carbonates is associated with qualitative differences in the samples studied.

Keywords: Minerals, Marble, Calcite, Dolomite, Limestone, Siderite, Thermal conductivity, Phonon-defect scattering, Temperature dependence

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1. Introduction

Calcium carbonate CaCO_3 and calcium magnesium carbonate $\text{CaMg}(\text{CO}_3)_2$ (dolomite) are the basis of many limestone rocks. Calcium carbonate is mainly represented by crystal structures such as calcite (sp. gr. *R3c*) and aragonite (sp. gr. *Pmcn*), less common is water-soluble vaterite (sp. gr. *P63/mmc*). Among natural carbonates, magnesite MgCO_3 and siderite FeCO_3 , isostructural to calcite, are also common. Marble is a limestone recrystallized at elevated temperature and pressure (metamorphosed), consisting mainly of calcite. The basis of dolomite marble is calcium-magnesium carbonate (sp. gr. *R3*).

The area of application of marble is extremely wide. Their use as a construction, finishing and facing material for bathrooms, swimming pools, fireplaces, etc. requires the presence of a certain heat flow and a corresponding temperature difference. The value of the latter is associated with such a thermophysical characteristic of the material as the thermal conductivity coefficient. Knowledge of the thermal conductivity values of rocks is necessary for the production of geophysical models. Thermal conductivity, related to the propagation rate of sound waves, for polycrystalline materials depends on the porosity and structure of grain boundaries and, thus, allows evaluating the mechanical characteristics of rocks.

The objective complexity of heat transfer processes does not allow to provide a sufficiently accurate a priori assessment of this characteristic, therefore experimental methods of determination is a priority. Studies [1–16] are devoted to the study of the thermal conductivity of natural carbonates. Theoretical calculations were provided in [17]. However, the publications we found provide the values of the thermal conductivity coefficient at room temperature or consider the behavior of the thermal conductivity of both rocks and crystalline calcite and dolomite at elevated temperatures. However, the low-temperature region is of practical and scientific interest, for example, for geophysical calculations in the Arctic. At the same time, experimental data of different authors on the thermal conductivity of marbles and other limestone rocks vary significantly, probably, mainly due to the qualitative diversity of the studied samples.

The purpose of this study was experimental investigation of the thermal conductivity coefficients of natural carbonate samples in a wide temperature range, from sub nitrogen to elevated. This study continues our measurements of the thermal conductivity of natural minerals and rocks [18–21].

2. Experimental

Natural samples listed in Table 1 and shown in Fig. 1 were used as research objects. Four metamorphosed samples (marbles № 1–4) and four sedimentary rock samples (№ 5–8) were examined.

X-ray phase analysis (XPA) was carried out using Bruker D8 Advance diffractometer ($\text{CuK}\alpha$ -radiation). The TOPAS software package was used to calculate the lattice parameters. The microstructure of the samples was assessed by optical microscopy using a microscope μVizor 103 and scanning electron microscopy (SEM) on an EVO 10 microscope (Zeiss, Germany) with LaB_6 cathode. Semi-quantitative chemical microanalysis (SEM-EDS) was performed using a Smart EDX spectrometer (METEK-Zeiss, USA & Germany).

Samples for thermal conductivity measurements were cut from homogeneous areas of the monoliths. For the temperature range of 50–300 K, samples in the form of parallelepipeds with the dimensions indicated in the Table 2 were used. For studies at elevated temperatures, samples were prepared in the form of tablets with a diameter of 15 mm and a thickness of 5 mm. It should be remembered that the indicated dimensions assume averaging of measurement results over a fairly large volume of samples. This distinguishes our method from the method used, for example by Merriman et al. [14] when studying samples with dimensions of ~1 mm.

Thermal conductivity coefficient in the temperature range 50–300 K was measured using the absolute stationary method of longitudinal heat flow. The experimental equipment and measurement technique are described in [23]. To ensure a flat heat front, a resistive heater that sets the measured temperature difference along the sample was glued to its end surface. The measurement error for the thermal conductivity coefficient value was not higher than $\pm 5\%$.

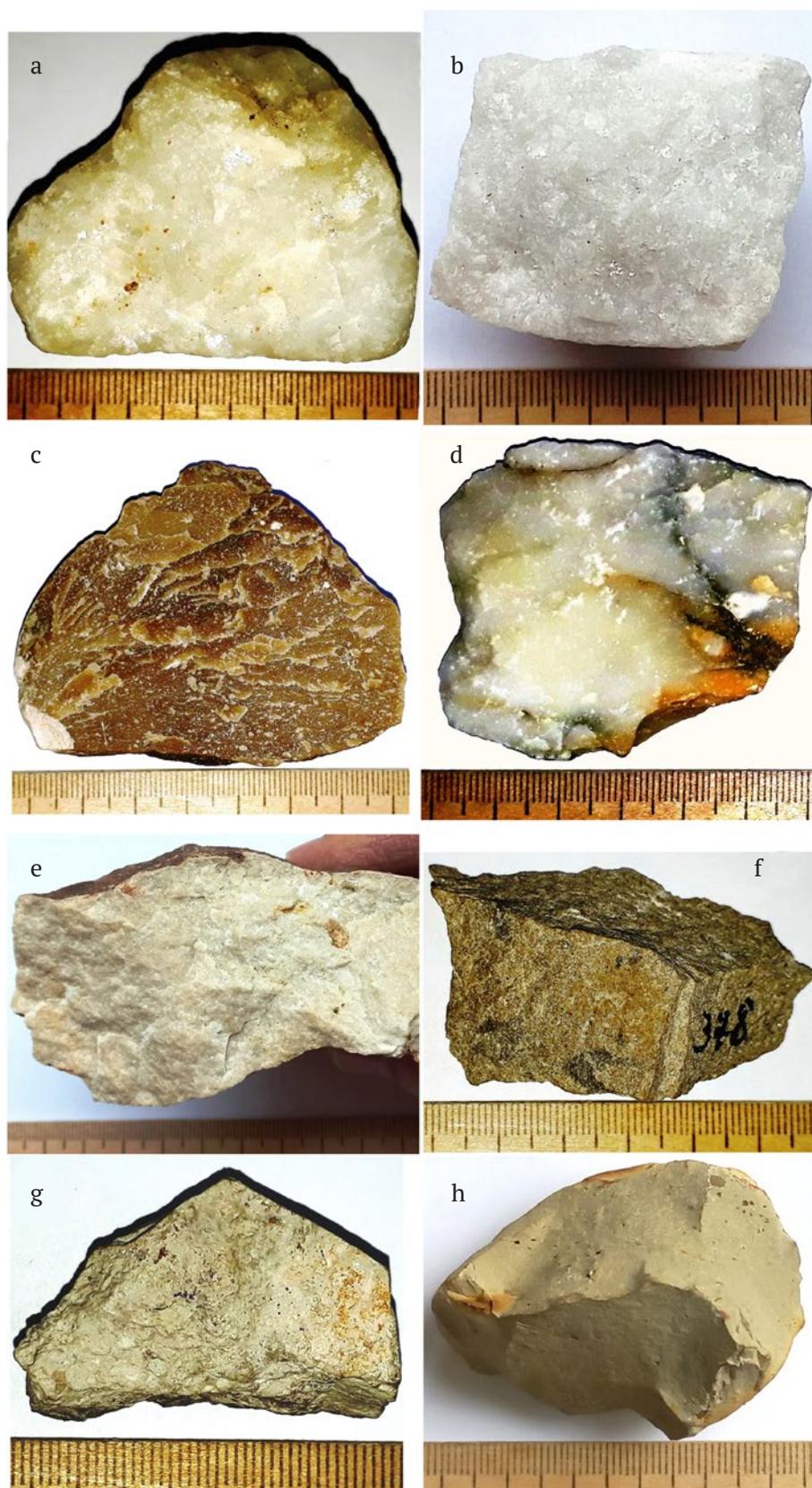


Fig. 1. Photographs of the studied samples: a – No. 1; b – No. 2 (marble); c – No. 3; d – No. 4; e – No. 5; f – No. 6; g – No. 7; h – No. 8

Table 1. Characteristics of the studied samples

No.	Name, location	Phase and chemical composition	Lattice parameters		Average grain size, microns
			a , Å	c , Å	
1	Dolomite marble, Ulan-Ude	Dolomite, $\text{CaMg}(\text{CO}_3)_2$	4.8142±0.0001	16.033±0.004	250
2	Marble, Slyudyanka, Dynamitny quarry	Magnesian calcite $\text{Ca}_{0.934}\text{Mg}_{0.07}\text{CO}_3$ + dolomite, $\text{CaMg}(\text{CO}_3)_2$	4.963±0.001	16.95±0.01	1000
			4.808±0.001	16.02±0.05	
3	Marble, Palanga	Calcite, $\text{Ca}_{0.96}\text{Mg}_{0.04}\text{CO}_3$	4.984±0.001	17.03±0.01	30
4	Marble, Greece, Kassandra Peninsula	Calcite, $\text{Ca}_{0.96}\text{Mg}_{0.04}\text{CO}_3$	4.990±0.001	17.01±0.01	20
5	Limestone, Ruza	Calcite	4.984±0.001	17.03±0.01	
6	Dense limestone, Tarusa ("Tarus marble")	Calcite, $\text{Ca}_{0.96}\text{Mg}_{0.04}\text{CO}_3$	4.985±0.001	17.04±0.01	40
7	Dolomite, Vazuza River	Dolomite $\text{CaMg}(\text{CO}_3)_2$ + quartz SiO_2	4.814±0.002	16.06±0.01	40
8	Siderite, Ruza [22]	Siderite, FeCO_3	4.705±0.003	15.45±0.01	5–10

Table 2. Geometric dimensions of samples in the form of parallelepipeds mm^3 and thermal conductivity coefficient values ($W/(m \times K)$) of the studied samples at different temperatures

T , K	No 1	No 2	No 3	No 4	No 5	No 6	No 7	No 8
50	15.2	10.5	9.4	8.23	8.67	4.76	2.30	1.17
100	9.37	7.83	6.72	5.84	5.38	4.42	3.33	1.47
150	7.06	6.14	5.15	4.61	4.15	3.88	3.49	1.67
200	5.89	5.14	4.22	3.91	3.48	3.40	3.39	1.80
250	5.21	4.52	3.62	3.51	3.07	3.04	3.25	1.87
300	4.72	4.06	3.24	3.22	2.76	2.79	3.08	1.90
350	4.3	3.7	3.0	3.0	2.5	2.6	–	1.9
400	4.0	3.4	2.7	2.8	2.4	2.4	–	2.0
450	3.7	3.2	2.6	2.6	2.3	2.2	–	2.0
500	3.5	3.1	2.5	2.4	2.2	2.1	–	2.0
550	3.3	3.0	2.4	2.3	2.1	2.1	–	2.0
573	3.3	2.9	2.4	2.3	2.1	2.1	–	2.0
Geometric dimensions of samples cut for measurements, mm^3								
	10.1× 9.4× 25	12.4× 10.7× 25	10.7× 9.6× 19	9.6× 8.1× 23	12.2× 13.7× 25	11.1× 8.7× 23	9.7× 9.7× 20	11.5× 12.5× 25

To study thermal conductivity coefficient in the range of 323–573 K, an ITλ-400 thermal conductivity meter was used with a measurement error within $\pm 10\%$.

3. Results and discussion

The studied samples were single-phase, with the exception of sample No. 2 (two-phase mixture of magnesian calcite with dolomite) and sample No. 7 (dolomite with a significant content of non-isomorphic impurities: about 3% quartz, traces of calcium phosphate). Using the phase

diagram of the CaCO_3 - MgCO_3 system [24] as a geothermometer, it can be stated that in two-phase sample No. 2 the magnesium content in the $\text{Ca}_{1-x}\text{Mg}_x\text{CO}_3$ solid solution corresponded to a metamorphization temperature of about 600° C.

Extrapolation of the limiting solubility curve of magnesium in the $\text{Ca}_{1-x}\text{Mg}_x\text{CO}_3$ solid solution, obtained by Golschmit and Hurd [24], up to room temperature provided the value $x \leq 10^{-4}$. Increased magnesium content in single-phase calcite samples (Table 1), several orders of magnitude higher than the equilibrium solubility of magnesium in a

solid solution in the $\text{CaCO}_3\text{-MgCO}_3$ system at low temperatures probably indicates the biogenic origin of these limestones [25].

Graphs of the temperature dependence of thermal conductivity coefficient $k(T)$ of studied samples are shown in Fig. 2. In numerical form, the measurement results $k(T)$ are shown in Table 2.

It can be seen that the obtained data varied widely, especially in the low temperature region. The dependence of thermal conductivity coefficient on temperature varied from monotonically decreasing, characteristic of single-crystal samples, to increasing, characteristic of highly disordered materials.

At room and higher temperatures, the thermal conductivity coefficient of all studied samples was below $5 \text{ W}/(\text{m}\cdot\text{K})$, which conditionally allows these materials to be classified as thermal insulators. The low hardness of the compounds (less than 4 according to Mohs), the characteristic cleavage of single crystals and the relatively low melting and decomposition temperatures indicate the weakness of interionic bonds, which negatively affects the efficiency of the heat transfer process.

The $k(T)$ points of dolomite sample No. 1 (curve 1) are located above all in Fig. 2. Over the entire temperature range studied, they were satisfactory (correlation coefficient $R = 0.9978$) approximated by a power function $k(T) = 190.8 \cdot T^{-n}$, where the exponent $n = 0.651$ is not much lower than the corresponding values, close to $n = 1$, for many dielectric single crystals with a perfect structure [26–28]. When the temperature was decreased to minimal $T = 50 \text{ K}$, the increase in growth rate $k(T)$ with a transition to an exponential dependence characteristic for crystals [29], or, conversely, a decrease in growth rates $k(T)$ with reaching the maximum were not observed. Probably, this maximum $k(T)$ characteristic of crystalline materials occurs at significantly lower temperatures. For samples of marble No. 4 and limestone No. 5, a similar approximating function has the form $k(T) = 67.08T^{-0.533}$ ($R = 0.9986$) and $k(T) = 97.333 \cdot T^{-0.623}$ ($R = 0.9967$) respectively. The coverage of such a wide temperature range by one function can probably be explained by the dominance of phonon scattering processes at grain boundaries.

The $k(T)$ graphs of marbles No. 2, No. 3, and, to a greater extent, limestone No. 6 (curves 2,

3 and 6 accordingly) demonstrate a decrease in growth rates when the temperature was decreased to $T = 50 \text{ K}$. For a low absolute value of thermal conductivity, this is a common circumstance for materials with partial structural disorder.

Quartz inclusions (sample no. 7, curve 7) into the dolomite matrix led to a sharp drop in thermal conductivity coefficient with a blurred maximum $k(T)$ with $k \rightarrow 0$ at $T \rightarrow 0$. The obvious reason for this is significant phonon-defect scattering.

The thermal conductivity of siderite demonstrated (curve 8) maximum disorder of the structure with the average phonon mean free path probably reaching the minimum possible value comparable to interstitial distances. The small sizes of crystal grains suggest the manifestation of intense phonon scattering due to the frequent intersection of grain boundaries by the heat flow. It should be noted that for the sample studied [22], the broadening of lines in the X-ray diffraction pattern indicates the size of the coherent scattering regions $D = 26 \text{ nm}$.

The comparison of the data obtained for dolomite (No. 1) and calcite (No. 4) marble samples with literature data based on the results of measuring the thermal conductivity of related materials is shown in Fig. 3.

It can be seen that the thermal conductivity coefficient of dolomite determined in our study

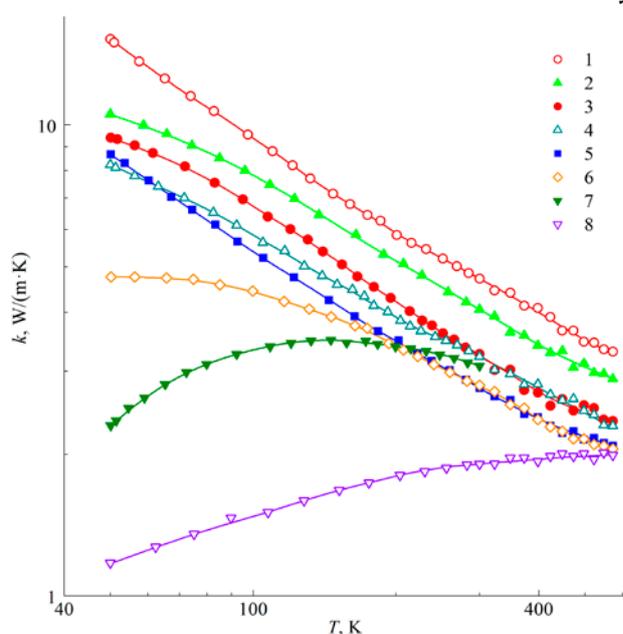


Fig. 2. Temperature dependences of thermal conductivity coefficients of samples: No. 1 (1); No. 2 (2); No. 3 (3); No. 4 (4); No. 5 (5); No. 6 (6); No. 7 (7); No. 8 (8)

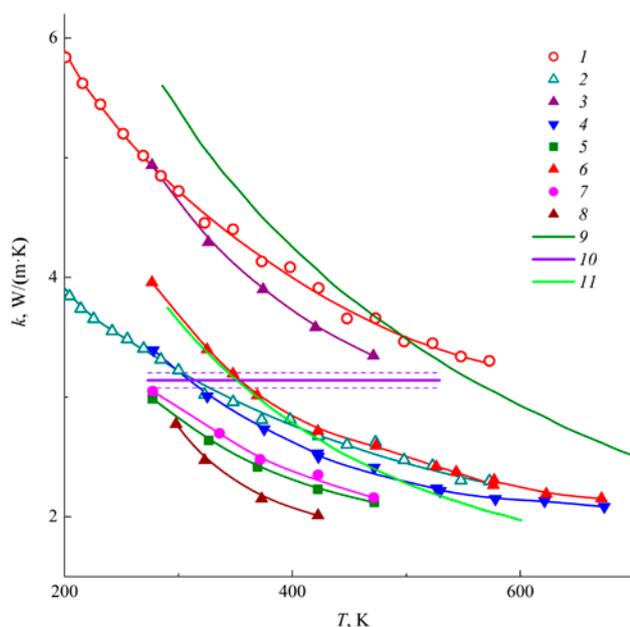


Fig. 3. Temperature dependences of thermal conductivity coefficients: sample No. 1 (1); sample No. 4 (2); dolomite [1] (3); calcite single crystal perpendicular to the c axis [1] (4); marble perpendicular to the layer [1] (5); single crystal of calcite along the c axis [1] (6); marble parallel to the layer [1] (7); marble [13] (8); dolomite, averaging for four samples [14] (9); dolomite [13] (10); calcite [14] (11)

is close to that obtained by Merriman et al. [14] (curve 9), which presented the result of averaging $k(T)$ for four mineral samples with an average grain size of 0.369 mm, skeletal density of 2.83 g/cm³ and porosity less than 1% and containing small quantities of iron (FeO = 0.18–0.42 wt. %) and manganese (MnO = 0.1 wt. %). Moreover, the total content of CaO + MgO in this mineral is 98.94–99.23 wt. % compared to ideal dolomite, and trace impurities (<0.1 wt. %) include oxides of Al, Na and Ti [14]. We should note the steepness of the dependence $k(T)$, when describing it by a power function $k \sim T^{-n}$ the value of the exponent in the high temperature region exceeds $n = 1$. Such a strong dependence is not typical for materials in a stable crystalline state [29, 30]. In terms of the absolute value of thermal conductivity coefficient, the $k(T)$ data of Birch and Clark [1] for dolomite (curve 3) with a density of 2.826 g/cm³ and average grain size of 0.01 mm also not significantly differ from our data. At the same time, the different degrees of steepness of graphs 1 and 3 should be noted.

It was also recorded [13] that there was virtually no dependence of thermal conductivity

coefficient on the temperature (horizontal line 10 in Fig. 3) of dolomite with a density of 2.70 g/cm³ and open porosity of 1–2%. Emirov et al. explained this feature by the presence of an amorphous component in the structure of the material.

Marbles studied by Birch and Clark [1] (curves 5 and 7) and Garcia [11] (curve 8), were significantly inferior by thermal conductivity to the marble sample No. 4 which we studied (curve 2). It is important to note that the obtained $k(T)$ graph for sample No. 4 at elevated temperatures occupies an intermediate position between the curves 4 and 6 for single-crystalline calcite according to [1].

Thus, in general, it can be stated that the thermal conductivity coefficient of crystalline dolomite is higher than that of calcite. The structures of these two minerals are similar to each other; dolomite differs by the substitution of half of the calcium cations with magnesium cations, which formed ordered layers located perpendicular to the c axis (Fig. 4). Usually, the substitution of some cations with the formation of a solid solution with isovalent isomorphism leads to a decrease in thermal conductivity. This fact has been documented in numerous examples for compounds with a fluorite structure [31]. However, the calcite-dolomite pair falls out of this pattern. The increased thermal conductivity of dolomite may be associated with a sharper increase in mechanical characteristics in relation to the increase in density during the transition from calcite to dolomite.

4. Conclusions

Thus, experimental data on the thermal conductivity of dolomite, calcite marbles and limestones in a wide temperature range - from sub nitrogen to elevated temperatures were obtained. In most cases, the thermal conductivity decreased in the specified range of materials. The revealed wide variations in the nature of the temperature dependence of thermal conductivity were associated with significant differences in the degree of disorder of the studied objects.

Contribution of the authors

The authors contributed equally to this article.

Conflict of interests

The authors declare that they have no known competing financial interests or personal

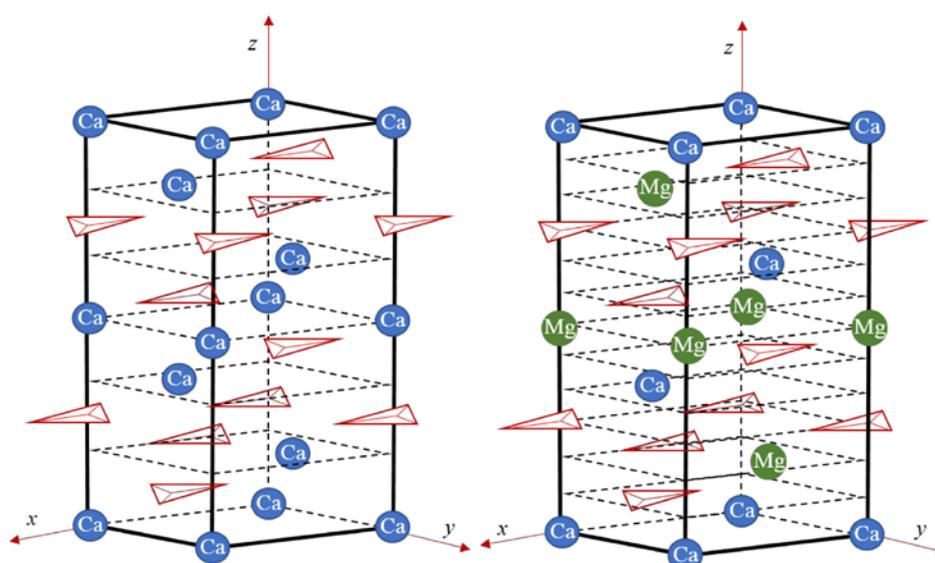


Fig. 4. Crystal structure of calcite (a) and dolomite (b): Red triangles indicate CO_3 groups [17]

relationships that could have influenced the work reported in this paper.

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