

KINETICS OF CARBON DIOXIDE MECHANOSORPTION BY Ca-CONTAINING SILICATES CO₂ release on heating of mechanically activated samples

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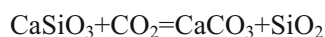
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Kinetics of mechanically induced CO₂ extensive sorption by silicate minerals (labradorite, diopside, okermanite, ghelenite and wollastonite) was considered. Mechanical activation of the silicates was carried out in a planetary mill in CO₂ at atmospheric pressure. Carbon dioxide was consumed by the silicates in the form of carbonate ions and its content in the samples after 30 min of mechanical treatment reached 3–12 mass% CO₂ depending on mineral composition. Equations that reasonably good represent kinetics of CO₂ mechanosorption by silicates were proposed. These equations enable to calculate mechanosorption coefficients that characterize the diffusivity of CO₂ into disordered silicate matrix under intensive mechanical impact. Thermal analysis of the mechanically activated silicates showed that there was positive correlation between temperature of complete carbonate decomposition and mechanosorption coefficient.

Keywords: carbon dioxide, kinetics, mechanical activation, mechanosorption, silicate minerals, thermal analysis

Introduction

It was revealed in previous work that prolonged grinding in air of calcium and magnesium containing silicates such as diopside CaMgSi₂O₆, okermanite Ca₂MgSi₂O₇, wollastonite CaSiO₃ and others resulted in extensive sorption of atmospheric carbon dioxide by the minerals [1]. Normally at ambient conditions the silicates (which are quite stable rock-forming minerals) are virtually inert to carbon dioxide. Mechanical activation (MA) extremely enhanced their reactivity with respect to CO₂ [2, 3]. Ca and Mg silicates selectively absorbed carbon dioxide from the environment under mechanical treatment in the form of carbonate ions. Carbon dioxide content in the silicates, as a result of prolonged grinding in air or intensive mechanical treatment in planetary mill in CO₂ atmosphere, reached more than 15 mass% CO₂ that was comparable with calcium content in the minerals. On this basis one might suggest occurring of chemical reaction between silicate and carbon dioxide producing silica and corresponding carbonate (e.g. calcite or aragonite). For example for wollastonite this reaction would be written as



However extensive CO₂ mechanosorption occurred alongside with silicate structure disordering and was not accompanied by formation of calcite or other carbonate minerals according to X-ray diffraction (XRD) data. Special experiments showed that the large

carbonate content in the mechanically treated silicates could not be explained by CO₂ surface chemisorption or by formation of amorphous calcium carbonate [2, 4, 5]. The obtained data suggested that CO₂ penetrated the structurally disordered silicate matrix and ‘dissolved’ in the particles volume with formation of carbonate-silicate phase as a result of deep mechanoabsorption. According to IR spectroscopic data this phase was similar to finely ground glass obtained by quenching of silicate melt wherein CO₂ was dissolved at magma state conditions ($T \sim 1800\text{--}2000$ K, $P \sim 1\text{--}2$ GPa) [1].

Grinding of silicates is a necessary and widely used operation in ore dressing, building materials and ceramics production. Mechanically induced sorption of carbon dioxide by silicate particles may influence their surface properties and, consequently, the effectiveness of flotation, hardening of binding materials, sintering. In addition, it relates to the problem of utilization of silica-based waste [6].

In principle, the revealed effect can be used for CO₂ removal from flue gas emissions by means of silicates and binding this greenhouse gas in an environmentally safe form. In this study kinetics of CO₂ mechanosorption by natural and synthetic Ca containing silicates (plagioclase, diopside, okermanite, ghelenite and wollastonite) using a laboratory planetary mill has been considered. Data on CO₂ release on heating of mechanically activated silicates using differential thermal analysis (DTA) and thermogravimetry (TG) have been presented.

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Experimental

Samples

Natural plagioclase, diopside, okermanite and synthetic ghelenite and wollastonite samples were used for experiments. Chemical composition of the minerals is given in Table 1. Plagioclase sample (fraction $-250+125 \mu\text{m}$) was from Gorodische deposit (Ukraine). It contained less than 1 mass% of magnetite as admixture. Plagioclases form discontinuous solid solutions from anorthite $\text{CaAl}_2\text{Si}_2\text{O}_8$ to albite $\text{NaAlSi}_3\text{O}_8$ by substitution of Ca and Al for Na and Si. According to $\text{CaO}/\text{Na}_2\text{O}$ percentage (Table) the plagioclase sample can be classified as labradorite [7].

Both diopside and okermanite samples (fractions $-200+125 \mu\text{m}$) were from Kovdor deposit (Kola Peninsula, Russia) and contained less than 1% of carbonates and micas as admixtures.

Ghelenite $\text{Ca}_2\text{Al}_2\text{SiO}_7$ and wollastonite CaSiO_3 were synthesized from reagent grade CaCO_3 , Al_2O_3 and amorphous silica. Before synthesis calcium carbonate was heated at 200°C for 24 h. Alumina and amorphous silica were annealed at 1000°C for 12 h. Stoichiometric mixtures of the components in both cases were ground in a Fritsch Pulverisette 2 laboratory mortar grinder for 10 h. After grinding mixtures were annealed at 900°C for 8 h and then at 1300°C for 12 h. Grinding for 10 h and annealing at 1300°C for 12 h were repeated 8 times. According to XRD and optical microscopy analyses the obtained substances were pure ghelenite and wollastonite (in the form of cyclowollastonite).

Methods

MA was performed in an AGO-2 planetary ball mill [8] at 40 g centrifugal factor. The initial sample mass was 10 g. The steel ball (5 mm in diameter) load to sample mass ratio was 20. Immediately before grinding, the air in the grinding vial was displaced by CO_2 from cylinder using higher density of carbon dioxide, and the vial was sealed hermetically. After 1, 2, 6, 10 and 20 min of MA about 1 g of sample was taken for analysis. After each sampling and every 2 min of MA the vial was repeatedly filled with CO_2 at $P=10^5$ Pa. According to chemical analysis of the

silicate minerals samples after MA for 30 min in CO_2 atmosphere the milling yield was within 1.5–2.0 mass% Fe.

Differential thermal analysis and thermogravimetry were performed in Ar at heating rate 10 K min^{-1} up to 1200°C by using Netzsch STA 409 instrument. X-ray diffraction (XRD) data were measured using DRON-2 equipment (CuK_α radiation). The carbon dioxide amount absorbed by silicates during MA was determined volumetrically using an AN-7529 gas analyzer. A weighed portion of the sample was calcined at 1200°C and the released CO_2 was absorbed by strontium chloride solution resulting in its acidation. The obtained solution was neutralized by electric current and the amount of absorbed CO_2 was determined coulometrically. The minor amount of carbonate present in natural minerals due to presence of carbonate minerals as admixtures was subtracted from measured CO_2 content of the mineral samples after MA. The specific surface area was measured using FlowSorb II 2300 (Micromeritics) analyzer by the low temperature nitrogen adsorption method.

Results and discussion

Kinetic analysis

Figure 1 shows carbonization degrees (mass% CO_2) of the studied silicates depending on MA time in CO_2 atmosphere. It is seen that CO_2 content in wollastonite, ghelenite and okermanite samples was similar at the same MA time. Diopside and particularly labradorite exhibit lower mechanically induced carbon dioxide sorption ability. These data are in agreement with previously revealed tendency: the larger the calcium content in mineral (Table 1) the higher its carbonization degree during grinding [1, 4]. According to XRD data in the course of MA crystal structure disordering of the samples increased. After 30 min of MA wollastonite sample (Fig. 2) and all other studied mineral samples were almost completely amorphous.

It followed from the slope of the curves (Fig. 1) that the intensity of carbonization process for all studied minerals gradually slowed down with increase of MA time. It should be noted that the rigorous consideration of mechanosorption kinetics is a complicated

Table 1 Chemical composition of initial samples (mass%)

Sample	SiO_2	Al_2O_3	CaO	Na_2O	K_2O	FeO	Fe_2O_3	MgO	TiO_2
Labradorite	53.7 ± 0.5	26.3 ± 0.20	10.80 ± 0.10	4.65 ± 0.05	0.45 ± 0.03	1.62 ± 0.05	0.61 ± 0.05	1.03 ± 0.05	0.28 ± 0.02
Diopside	49.7 ± 0.5	1.34 ± 0.05	24.50 ± 0.15	0.43 ± 0.05	0.19 ± 0.02	2.88 ± 0.05	–	16.10 ± 0.10	0.48 ± 0.05
Okermanite	42.4 ± 0.5	3.60 ± 0.05	34.5 ± 0.20	1.80 ± 0.05	0.10 ± 0.02	2.75 ± 0.05	–	9.40 ± 0.10	–
Ghelenite	21.92	37.18	40.90	–	–	–	–	–	–
Wollastonite	51.72	–	48.28	–	–	–	–	–	–

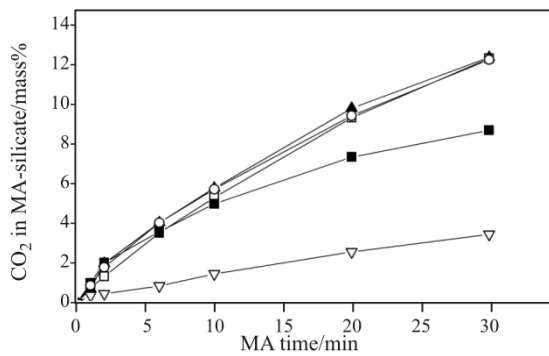


Fig. 1 Carbonization degrees (mass% CO₂) of silicate minerals depending on MA time in CO₂ atmosphere: ▲ – okermanite, ○ – wollastonite, □ – ghelenite, ■ – diopside, ▽ – labradorite

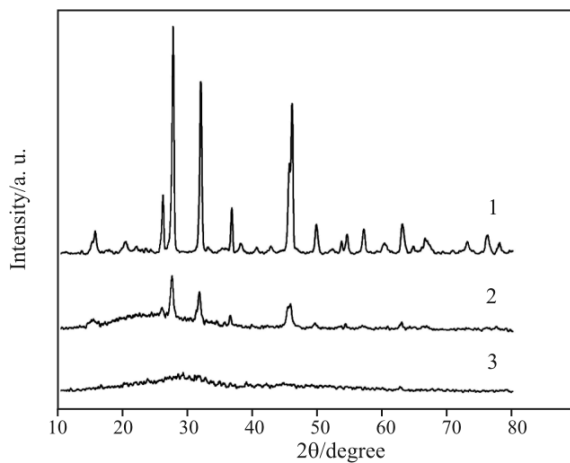


Fig. 2 XRD patterns of wollastonite samples: 1 – initial sample; 2 and 3 – after 2 and 30 min of MA in CO₂ atmosphere, respectively

problem due to variety of physical–chemical processes involved in mechanical action upon solid–gas system and their non-equilibrium nature [8–11]. Mechanism and kinetics of mechanically induced absorption of inert gases by metals and chemisorption of hydrogen onto quartz particles surface were studied in [9, 12], respectively. In the former case inert gases physically ‘dissolved’ in the surface layer of metal under mechanical impact. In the latter one hydrogen molecules chemically reacted with free radical centers on the surface of quartz. In the course of MA of silicates in CO₂ atmosphere carbon dioxide molecules both penetrated the structurally disordered mineral particles and chemically reacted with oxygen anions forming CO₃²⁻ groups [1–4]. Under these circumstances it is reasonable to determine the basic factors that influence mechanosorption and to apply a simplified model.

Two modes of gas sorption were revealed during study of quartz vibro-milling in H₂ atmosphere [12]. During initial stage of grinding mechanical energy was

mainly spent for fresh surface formation. During this so called cleavage mode hydrogen molecules were chemisorbed on active centers of the newly formed surface. The rate of gas sorption in the cleavage mode was in direct proportion to the rate of active centers growth and consequently to the rate of surface area increase. As the total surface area *S* rised during grinding, portion of active centers arising due to friction of particles surface increased (so called friction mode). In the friction mode the rate of gas sorption depended on the surface area value instead of the surface area time derivative as in the cleavage mode. The total rate of gas sorption *W* can be expressed as [12]:

$$W = W_{cl} + W_{fr} = a(dS/dt) + f_{fr}(S) \quad (1)$$

where *W*_{cl} and *W*_{fr} are the rates of gas sorption induced by cleavage and friction, respectively; *a* – coefficient that is proportional to the active centers concentration on the fresh surface. Contribution of friction to the total rate *W*_{fr}=*f*_{fr}(*S*) depends not only on the surface area but also on type of grinding, nature of gas and solid, and other factors. The analytical form of *f*_{fr}(*S*) is unknown. Using this general scheme we can consider carbon dioxide mechanosorption by the silicate minerals.

A typical change of mineral specific surface area during MA is shown in Fig. 3 by the example of wollastonite. The curve in Fig. 3 can be divided into two parts. During the first 1–2 min of MA the specific surface area *S*_{sp} rapidly increased. Then *S*_{sp} gradually decreased due to aggregation processes and turned to quasi-steady state. It is reasonable to assume that during the first 2 min of MA mechanosorption occurred mainly in the cleavage mode and partially in the friction one. After maximum (more than 2 min of MA) CO₂ sorption was virtually totally controlled by friction. During the first 1–2 min of MA in CO₂ atmosphere silicate particles with carbonized thin surface layer were formed. At this stage (the cleavage mode) the intensity of CO₂ sorption was the highest one (Fig. 1). Then dispersion, i.e. reducing of particles size and growth of surface area, stopped due to the

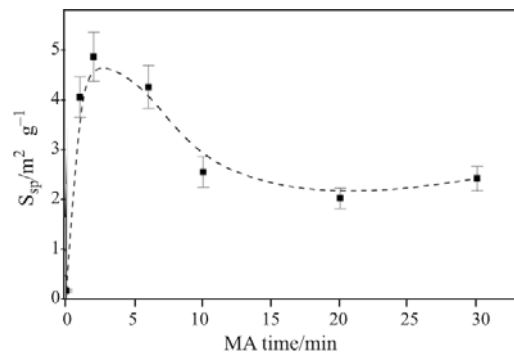
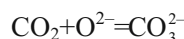


Fig. 3 Specific surface area of wollastonite depending on MA time in CO₂ atmosphere

size factor and aggregation processes [8, 9] (Fig. 3). Sorption of carbon dioxide by the silicates became slower at this stage because CO₂ molecules diffused inside the structurally disordered particles through the outer carbonized layer. It should be noted, that despite the notable decrease of surface area after 2 min of MA (Fig. 3) the rate of CO₂ sorption slowed down to only a small extent (Fig. 1). This is evidence that after 2 min of MA sorption mainly occurred in the friction mode. In IR spectra of MA-silicates (not shown) in 2370–2350 cm⁻¹ region there were no bands corresponding to physically adsorbed CO₂ or to carbon dioxide dissolved in molecular form [1–4]. Thus under intensive mechanical treatment resulting in accumulation of defects, deformation and break of bonds not only on the surface but also in the bulk of silicate particles the rate of chemical interaction



was high and the mechanosorption process as a whole was diffusion controlled.

Consider CO₂ mechanosorption by a silicate at MA time more than 2 min that is in the friction mode. Equation (1) can be rewritten in the form

$$W = W_{\text{fr}} = f_{\text{fr}}(S) \quad (2)$$

Mechanosorption rate is determined as $W = dM/d\tau$, where M – total mass of CO₂ absorbed by a silicate, S – total surface area of a solid. Applying Fick's laws of diffusion in semi-infinite solid approximation [13] one can derive

$$W = f_{\text{fr}}(S) = dM/d\tau = K_{\text{ms}}(S^2/M) \quad (3)$$

where K_{ms} – mechanosorption coefficient (g² m⁻⁴ min⁻¹). Actually K_{ms} is a semi-empirical parameter that characterises mechanosorption ability of silicate with respect to carbon dioxide. K_{ms} depends on carbon dioxide diffusivity and concentration of CO₂ (present in the form of carbonate groups) in the outer layer of silicate particles [13].

Equation (3) can be transformed taking into account changing of S in the course of MA. M and S can be written in the form

$$M = 0.01Cq/(1-0.01C) \quad (4)$$

$$S = S_{\text{sp}}q/(1-0.01C) \quad (5)$$

where q – initial mass of mineral (g), C – carbonization degree expressed as mass% CO₂ in MA-mineral, S_{sp} – specific surface area of MA-mineral (m² g⁻¹). One should bear in mind that both C and S_{sp} depend on MA time. After substitution of Eqs (4) and (5) in Eq. (3) and some rearrangement one can derive

$$-\ln(1-0.01C) = 10^2 K_{\text{ms}} \frac{S_{\text{sp}}^2}{C} d\tau \quad (6)$$

Integration of Eq. (6) from $\tau=2$ min to $\tau=t$ min (the friction mode) leads to

$$F(t) - F(2) = 10^2 K_{\text{ms}} \int_2^t \left(\frac{S_{\text{sp}}^2}{C} \right) d\tau = 10^2 K_{\text{ms}} \Theta(t) \quad (7)$$

where

$$F(t) = -\ln(1-0.01C(t)) \quad (8)$$

$$\Theta(t) = \int_2^t \left(\frac{S_{\text{sp}}^2}{C} \right) d\tau \quad (9)$$

For calculation of $\Theta(t)$ experimental values of S_{sp}^2/C were fitted by least-squares method using the following expression

$$S_{\text{sp}}^2/C = b_0 + b_1 \exp(-b_2\tau) + b_3 \exp(-b_4\tau) \quad (10)$$

A typical agreement between the experimental values of S_{sp}^2/C and the calculated ones using Eq. (10) is shown for wollastonite as the example in Fig. 4. Using Eq. (10) $\Theta(t)$ can be calculated in the form

$$\Theta(t) = b_0(t-2) - \frac{b_1}{b_2} [\exp(-b_2t) - \exp(-2b_2)] - \frac{b_3}{b_4} [\exp(-b_4t) - \exp(-2b_4)] \quad (11)$$

For wollastonite [$F(t) - F(2)$] values obtained by substitution of experimental $C(t)$ values to Eq. (8) plotted vs. $\Theta(t)$ calculated using Eq. (11) are shown in Fig. 5.

Squares in Fig. 5 from left to right correspond to 2 (point of origin), 6, 10, 20 and 30 min of MA, respectively. Provided that K_{ms} in Eq. (7) is constant points in Fig. 5 would display linear dependence with slope being equal to $10^2 K_{\text{ms}}$. However from Fig. 5 it follows that mechanosorption coefficient grew with increase of MA time. It can be explained by the following reason. In the course of MA due to accumula-

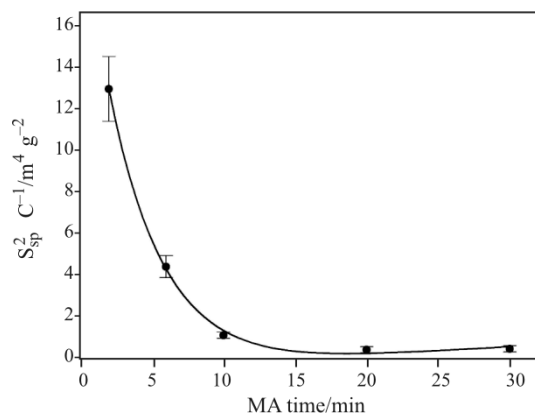


Fig. 4 ● – Experimental values of S_{sp}^2/C for wollastonite and — – approximation curve according to Eq. (10)

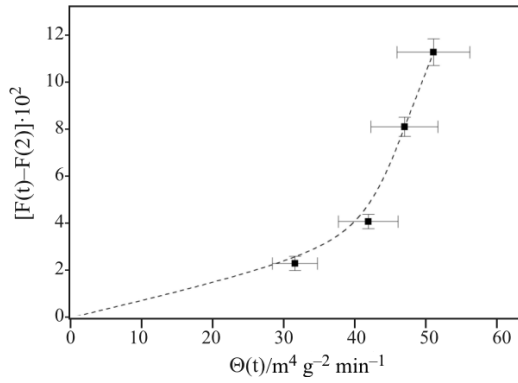


Fig. 5 $[F(t)-F(2)]$ values depending on $\Theta(t)$ for MA of wollastonite (see text for details)

tion of defects the silicate mineral structure disordering substantially increased (Fig. 2) thereby facilitating diffusion process.

In order to take into account the growth of mechanosorption coefficient with increase of MA time K_{ms} can be inserted into Eq. (7) in the form

$$K_{ms}(\tau)=k_0+k\tau \quad (12)$$

where k_0 and k are empirical parameters, τ is MA time, min. Substitution of Eq. (12) in Eq. (6) and integration yield

$$F(t)-F(2)=10^2(k_0\Theta(t)+k\psi(t)) \quad (13)$$

where

$$\psi(t)=\int_2^t \left(\frac{S_{sp}^2}{C} \right) \tau d\tau \quad (14)$$

$\Psi(t)$ can be easily calculated after substitution of Eq. (10) in Eq. (14). For evaluating of k_0 and k it is convenient to introduce $\Phi(t)$ and $\Omega(t)$:

$$\Phi(t)=10^{-2} \frac{F(t)-F(2)}{\Theta(t)} \quad (15)$$

$$\Omega(t)=\frac{\Psi(t)}{\Theta(t)} \quad (16)$$

$\Phi(t)$ values vs. $\Omega(t)$ are plotted in Fig. 6. Straight line in Fig. 6 corresponded to linear least-square fit of the points. The slope and intercept of the line are equal to k_0 and k in Eq. (13), respectively.

For wollastonite mechanosorption coefficient was expressed in the form

$$K_{ms}(\tau)=-(9.1\pm 0.9)\cdot 10^{-6}+(4.4\pm 0.4)\cdot 10^{-6}\tau \quad (17)$$

$(g^2 m^{-4} min^{-1})$

Note that extrapolation of Eq. (17) beyond the range of points plotted in Fig. 6, corresponding to time interval from 6 to 30 min of MA may lead to unreasonable results. Calculation of carbonization degrees C in the sampling points (6, 10, 20 and 30 min

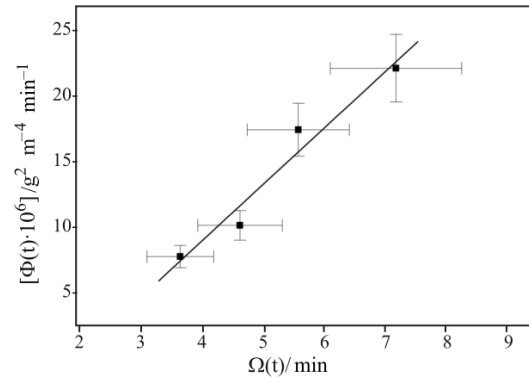


Fig. 6 $\Phi(t)$ values depending on $\Omega(t)$ for MA of wollastonite. From left to right squares corresponded to 6, 10, 20 and 30 min of MA, respectively (see text for details)

of MA) from obtained k_0 and k values using Eqs (11)–(14) showed that standard deviation between the experimental (Fig. 1) and the calculated values was 0.5 mass% CO_2 . So in spite of the simplified approach the suggested semi-empirical equations reasonably good described CO_2 mechanosorption kinetics for wollastonite. Similar agreement was achieved for the other studied minerals.

Thermal analysis of MA-silicates

Consider processes occurring during heating of MA-samples by the example of ghelenite. Figure 7 shows DTA, DTG and TG curves of ghelenite sample after MA in CO_2 atmosphere for 30 min. Small endothermic effect at $100^\circ C$ corresponded to removal of minor amount of adsorbed water present in the sample due to the contact with air during MA experiment. At elevated temperatures according to TG and DTG data there were two main steps of carbonate decomposition. Broad DTG peak at $540^\circ C$ corresponded to the first step of CO_2 release. According to the literature data [14, 15] this step was likely evolving of CO_2 due to decomposition of CO_3^{2-} groups related to surface carbonation. Dehydroxylation is also typical of this temperature [14, 15].

According to TG data the major part of carbonate decomposed during the second step in $900-1100^\circ C$ range. This step was accompanied by pronounced endothermic effect at $970^\circ C$. As was mentioned above after 30 min of MA all mineral samples including ghelenite one were amorphous. According to XRD data (not shown) CO_2 release in $900-1100^\circ C$ range was accompanied by crystallization of ghelenite. So, endothermic effect of carbonate decomposition and exothermal effect of crystallization overlapped. It explains bending of DTA, TG and DTG curves in $970-1100^\circ C$ range. Complete removal of carbon dioxide from MA-ghelenite according to TG data was achieved at $1100^\circ C$ (Fig. 7). Similar processes occurred during heating of the other MA-silicates.

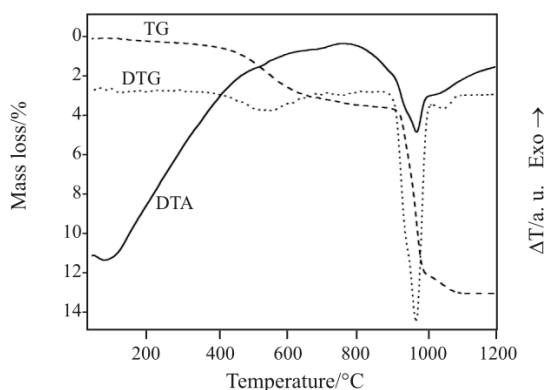


Fig. 7 DTA, TG and DTG curves of the ghelenite sample after MA in CO₂ atmosphere for 30 min

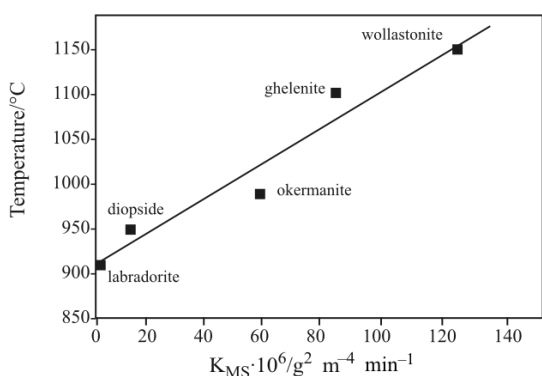


Fig. 8 Temperatures of complete CO₂ removal from the mineral samples after MA in CO₂ atmosphere for 30 min plotted vs. mechanosorption coefficients K_{ms} . For all minerals K_{ms} values corresponded to 30 min of MA

In Fig. 8 temperature of complete CO₂ removal from MA-samples after 30 min of MA was plotted vs. mechanosorption coefficient corresponding to 30 min of MA. Temperature of complete CO₂ removal was determined for each MA-mineral as the first TG curve point following which mass loss was no longer observed. It follows from Fig. 8 that there is obvious positive correlation between K_{ms} values and the temperature of complete carbonate decomposition on heating.

Conclusions

The suggested equations can be used for simplified kinetic analysis of mechanically induced CO₂ absorption by silicates. Mechanosorption coefficient K_{ms} can serve as semi-empirical parameter characterizing silicate ability to absorb carbon dioxide in the course of MA. One should bear in mind that K_{ms} depends on type of mill, mill load, and MA time. The comparison of K_{ms} values determined for different minerals is

valid if only MA is carried out at the same conditions. Thermal analysis of MA-samples revealed strong correlation between temperature of complete carbonate decomposition and K_{ms} values. It indicates that the higher CO₂ mechanosorption ability of silicate the stronger carbonate is bound in silicate matrix.

Acknowledgements

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