

SH-Synthesis of Nanostructured Materials Based on $\text{SiO}_2+\text{Al}+\text{CaSiO}_3$ with Wollastonite after Ultrasonic Treatment

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Abstract

This paper presents the results of a complex study of the structure and properties of wollastonite after ultrasonic treatment (UST) and its effect on the development of the process of technological combustion (SHS) of systems based on quartz, the structure and properties of synthesized composition materials. Ultrasonic treatment of wollastonite of the brand FW100 was carried out in water and 5% aqueous solutions of different alcohols and acids at 40 and 100 kHz as well as at simultaneous UST action of 40 and 100 kHz during 20-60 minutes. In the presence of different organic modifying additives during UST there takes place not only milling of wollastonite particles and erosion of their surface but also different complex structural changes on the surface of particles and their capsulation into colloidal conglomerates. All the structural changes taking place in wollastonite particles under the conditions of UST result also in the change of the chemical activity of the material, which effects the thermal characteristics of the combustion process (SH-synthesis) of the systems containing UST modified wollastonite in the charge mixture. It is stated that, when introducing wollastonite after ultrasonic treatment into SHS-charge, there takes place a more complete conversion of initial components in the combustion process. The greatest amount of corundum and nanocrystalline silicon is formed, when using wollastonite treated in water, aqueous solution of polyvinyl alcohol and acetylsalicylic acid. The synthesized samples are characterized by high stable values of strength (up to 73 MPa) and indices of electric insulation properties.

Introduction

When producing nanocomposition materials with high indexes of strength, crack resistance and other physico-mechanical properties under the conditions of self-propagating high temperature synthesis (SHS), i.e. technological combustion, nanopowders of oxide compounds are introduced into the composition of a charge mixture [1]. Mechanochemical treatment in mills with a dynamic action is one of the methods allowing to obtain ultra disperse powders with particles of a nanostructure [2]. Another effective method of changing both dispersity and structure of the surface layer of particles is ultrasonic treatment of powders in aqueous medium [3]. The use of ultrasound allows to obtain different emulsion systems, clean the surface of workpieces, disperse metallic, oxide, polymer and other systems up to production of nanomaterials. The main mechanisms of ultrasonic dispersion of powder materials are cavitation and flows initiated in a working fluid

due to collapse of cavitation bubbles [4]. The effect is specified by the fact that it takes the cavitation bubbles less than nanoseconds to get collapsed [5], the cooling rates exceeding 10^{11} K/s. Also, bubble explosions result in initiation of flows the velocity of which reaches 150 m/s. In a shock wave propagating from the point of collapse, the maximum amplitude of pressure can reach several thousand kilogram-forces per a square centimeter. The shock wave is followed by the wave with a negative pressure which exerts a stretching effect resulting in destruction of particles. Erosion of the surface and destruction of particles under the effect of ultrasound lead to the increase in the chemical activity of their surface and acceleration of interaction processes of the material particles with the medium of treatment [6, 7].

In this work, wollastonite was subjected to ultrasonic treatment. Wollastonite is one of the promising minerals in production of composition systems including those which contain objects of a nanoscale level [8]. A needle form of wollastonite grains al-

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lows to obtain a rigid framework which considerably improves the strength of products. When splitting wollastonite crystals, grains with a needle form are produced. At present, wollastonite is one of the main mineral fillers used in different branches of industry. It imparts qualitatively new useful properties to various materials and products or allows to improve these properties [9].

Thus, at present, a great attention is paid to the development of technologies and practical recommendations on production and use of heat resistant wollastonite containing technical ceramics of different purposes. SH-synthesis is one of the technological approaches to solution of this task – production of a new generation of heat resistant as well as heat insulation and refractory materials. Different methods of preactivation of charge mixtures provide the increase in the reactivity and dispersity of powders as well as formation of laminated nanosize structures. All this in complex influences the kinetics and thermodynamics of the combustion process and, consequently, the reaction products [1, 10, 11].

Thus, it follows from the analysis of literature data that wollastonite is one of the promising materials in production of different composition systems. Mechanochemical treatment extends the possibilities in creation of such systems, in particular, under the conditions of technological combustion. Considering that ultrasonic effect is a variety of mechanochemical action it is, undoubtedly, of great interest to state the regularities of the change in the structure and properties of wollastonite under the conditions of its ultrasonic treatment. It is necessary to determine the most effective conditions for modification of the treated powder and for carrying out SH-synthesis for production of strong ceramic composition systems of different purposes.

Materials and Methods of Investigation

To carry out SH-synthesis, we used quartz sand of Kuskuduk deposit with the content of quartz equal to 81.3%. Besides, it contains up to 18.7% of microcline $\text{K}(\text{Si}_3\text{Al})\text{O}_8$ as well as different elements saturating the surface and particles dissolved in the volume. According to the results of spectral analysis, it contains from 0.1 to 1.0% of iron, magnesium, calcium, sodium. Aluminium of the brand APV in a stoichiometric ratio to quartz was used as a reducing agent. The second mineral component of the charge mixture was wollastonite powder FW100 containing impurities of quartz in the amount of ~5%. The powder particles have a needle form with the size from 1 to 50 μm .

Wollastonite was subjected to ultrasonic treatment in aqueous medium with a soluble modifying

additive in the amount of 10%. Piezoelectric resonance adjusted transducers with the frequency of 40 and 100 kHz and power of 50 W were used as electro-acoustic radiator. Ultrasonic treatment was carried out with variation of time (during 20, 40 and 60 minutes) and frequency of action. The treatment was carried out at both separate and simultaneous action of low and high frequencies of ultrasonic range. The latter is more advantageous as cavitation bubbles appearing at low frequencies gain great acceleration under the action of hydrodynamic flows created by ultrasonic vibrations of high frequency. Glycerin – $\text{C}_3\text{H}_5(\text{OH})_3$, polyvinyl alcohol – $(\text{C}_2\text{H}_3\text{OH})_n$, aqueous silicic acid – $\text{H}_2\text{SiO}_3 \cdot n(\text{H}_2\text{O})$ and salicylic acid – $\text{C}_7\text{H}_6\text{O}_4$ served as modifying additives to aqueous medium during UST of wollastonite particles.

When moulding cylindrical samples with the diameter of 20 mm and height of 20-25 mm for SH-synthesis, natural silica gel from the hydro volcano (Karaganda region) in the amount of 5% was used. According to the results of X-ray phase analysis, the phase compositions of natural silica gel were as follows: quartz SiO_2 – 27.7%; clinoclhorine $2\text{M}-\text{Al}_2\text{Mg}_5\text{Si}_3\text{O}_{10}(\text{OH})_8$ – 14.9%; illite $1\text{M} - \text{K}_{0.7}\text{Al}_2(\text{Si},\text{Al})_4\text{O}_{10}(\text{OH})_2$ – 39.6%; albite $\text{Na}(\text{AlSi}_3\text{O}_8)$ – 17.6%.

Technological combustion was carried out in a muffle furnace with the pre-determined temperature of about 900 °C. The combustion temperature was measured by a pyrometric thermometer “RaytekRaynger 3i”. The sample temperature was recorded during the SH-synthesis with the help of thermocouples.

Experimental Results and Discussion

A complex investigation of the structure and properties of wollastonite after ultrasonic treatment and its effect on the development of the technological combustion process of the systems based on quartz, the structure and properties of the synthesized composition materials was carried out. After US-treatment we determined dispersity, bulk density of the powder, carried out electron-microscopic investigations of the particles and measured their dielectric permittivity. The dispersity data of the powder after US-treatment in water (Table 1) indicate the change of the structure. Dispersity of the powder decreases with both the time and frequency of US-action, the greatest effect is registered under simultaneous action by frequencies 40 and 100 kHz.

Electron-microscopic investigations give a clear view of the change in dispersity and morphology of particles after US-treatment of wollastonite. The morphology of particles was studied on the transmission electron microscope Jem-100CX; U-100kv.

Table 1

Dispersity of wollastonite powder (brand FW 100) depending on the frequency and time of US-treatment in water

Frequency of UST, kHz	Time of UST, min	Percentage, %					
		Dispersity, μm					
		0-11.6	11.6-23.2	23.2-46.4	46.4 -56.0	58-81.2	81.2-92.8
0	0	74	19				7
40	40	78	18			4	
40	60	82		13	4	1	
100	40	79	19		2		
100	60	80	16			4	
40+100	40	87	9	3	1		
40+100	60	89	10	1			

In the presence of different organic modifying additives in the course of UST there takes place not only milling of wollastonite particles but also erosion of their surface as well as different complex structural changes on their surface and capsulation into colloidal conglomerates. After US-treatment of glycerine in aqueous solution, highly disperse particles and their aggregates are encapsulated into quite dense polymer films shown in Fig 1.

After treatment in aqueous solution of polyvinyl alcohol the mass and size of particles increased. They are encapsulated into an organic film. It should be noted that during UST there takes place separation of wollastonite particles into layers, they preserve a needle form but the ratio of the length to the diameter of the particle significantly changes. This fact is most vividly presented after treatment in the presence of salicylic acid in Fig. 1.

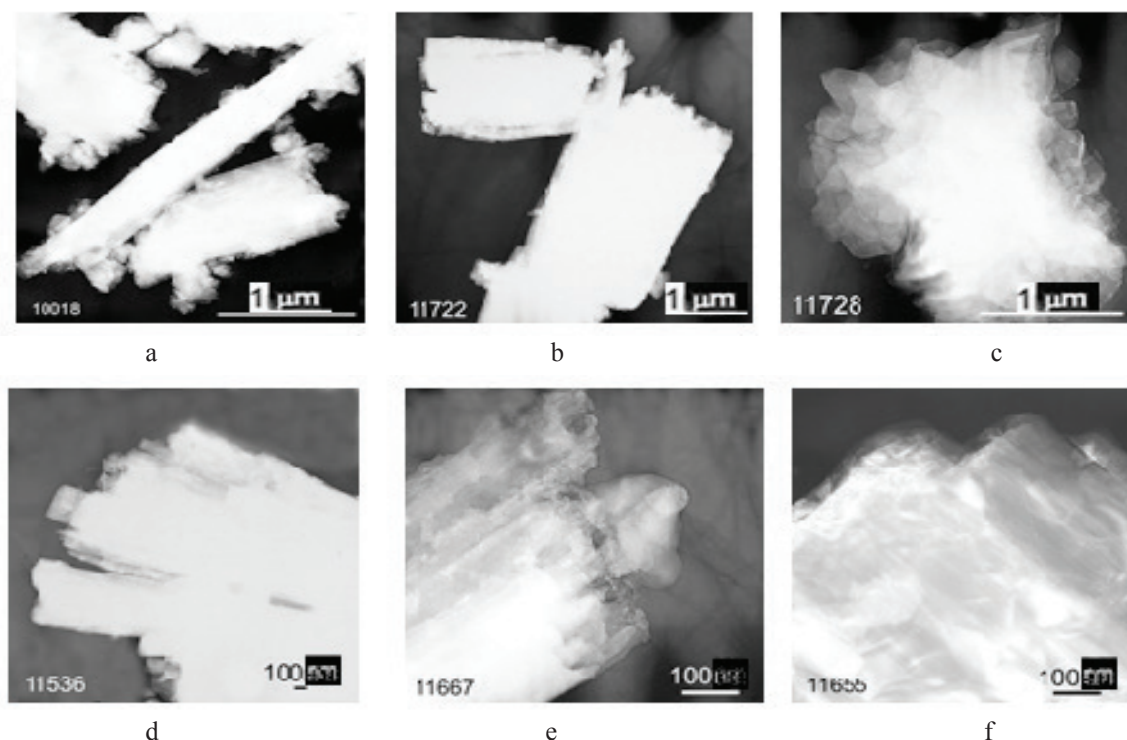


Fig. 1. Electron-microscopic pictures of wollastonite in the initial state (a) and after US-treatment during 40 minutes at 40 kHz in water (b), in aqueous solution of glycerine (c), salicylic (d) and silicic acid (e), polyvinyl alcohol (f).

The most significant changes in the morphology of wollastonite particles are observed after simultaneous US-treatment at 40 and 100 kHz. After

treatment in solution of glycerine, highly disperse particles and their aggregates are encapsulated into rather dense polymer films. When using polyvinyl

alcohol in the medium of treatment, alongside with grinding of wollastonite particles, there takes place intensive destruction of polyvinyl alcohol to carbon, the particles of which are present both independently and on the surfaces of wollastonite. Microdiffraction patterns indicate the presence of a great number of aggregates of small particles (Fig. 2b).

In the course of treatment in solution of salicylic acid and double action of UST at 40 and 100 kHz, alongside with the grinding of particles, the effect of their capsulation increases (Fig. 2c). When using silicic acid, this effect becomes more significant. The microdiffraction patterns indicate the presence of a great share of the amorphized substance (Fig. 2d).

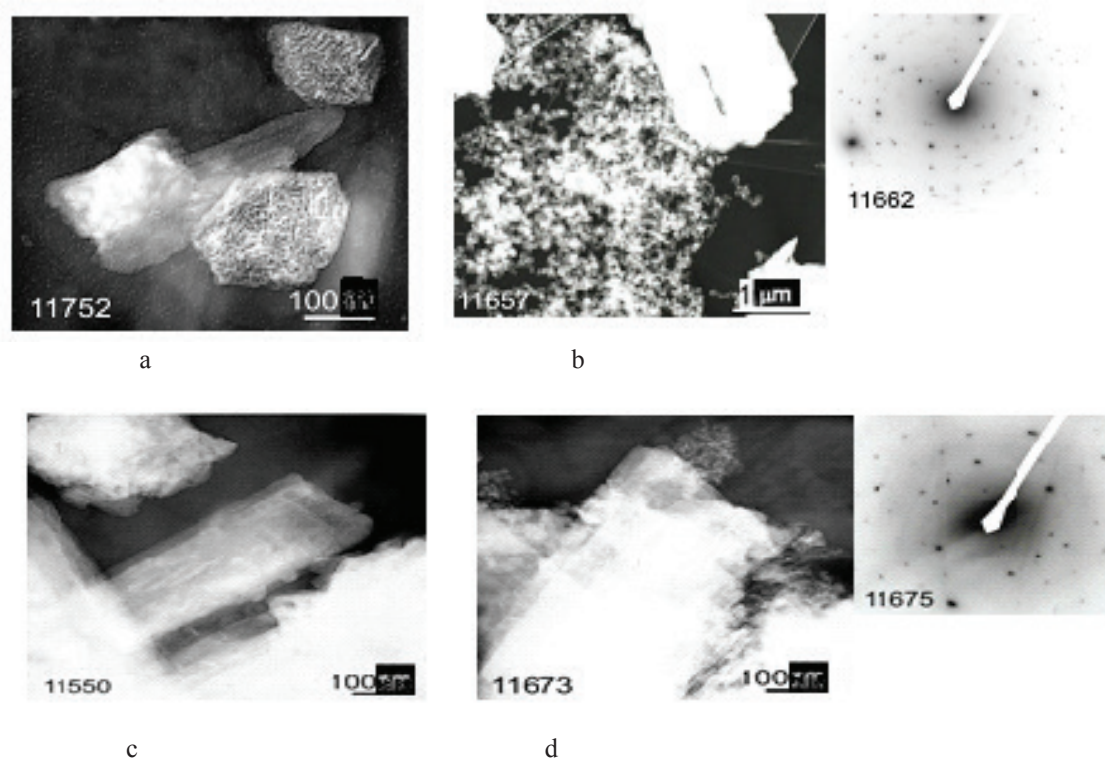


Fig 2. Electron-microscopic pictures of wollastonite, after US-treatment at double action of 40 and 100 kHz in an aqueous solution of glycerine (a), polyvinyl alcohol (b), salicylic acid (c) and silicic acid (d).

With the increase in frequency of US-treatment the size of crystallites in all the studied cases, except treatment in pure water, decreases. However, in the change of microdistortions of the crystalline lattice one can not observe a clear dependence on the treatment conditions, this can be related to different intensity and the degree of interaction of the modifying component in the aqueous medium with the surface of a wollastonite particle. Most probably, in the course of active chemical interaction of a modifier with wollastonite the flow of defects onto the surface (to the field of reaction) gets intensified, as a result, the degree of the lattice distortions decreases, this is registered, when using salicylic and silicic acids as modifiers. What is important here is that the presented results indicate the fact of structural changes in the ultrasound material being treated.

During UST there takes place not only grinding of wollastonite particles, formation, accumulation and

redistribution of defects in the volume of particles, erosion of their surface and capsulation into colloidal conglomerates but also more complex structural changes on the surface and volume of the particles being treated. Wollastonite refers to dielectrics and dielectric permittivity is a structure sensitive characteristic of the material. The value of dielectric permittivity is determined by polarization of dielectrics, i.e. limited displacement of bound charges in the dielectric or the turn of electric dipoles under the action of external electric field. Polarization does not change the total charge in any macroscopic volume inside a homogeneous dielectric. However, it is accompanied by appearance of bound electric charges with some surface density on its surface. Hence, all the structural changes taking place in the course of US-treatment of different materials: accumulation and redistribution of defects, break of bonds and formation of compounds on the surface of milled

particles with participation of modifying additives – must effect the index of dielectric permittivity. And it can serve as a characteristic of the new state of particles after US-treatment. The performed measurements of a relative dielectric permittivity of the material under study showed that if in the initial state wollastonite has $\varepsilon = 2.4$, after UST in water at the frequency of 100 kHz it decreases to 2.1. But the state of the material stabilizes.

The change of frequency, time and medium of US-treatment results in the change of the values of dielectric permittivity. The regularities of the change in the value ε are determined by concrete conditions of UST, but practically in all cases the presence of a

dissolved substance in water leads to the increase in the values of dielectric permittivity of wollastonite powder. This indicates the formation of particles of structural elements which undergo polarization in the electric field. After treatment at 40 kHz a stable increase of dielectric permittivity to 2.6 is observed after US-treatment in the presence of silicic acid, and the highest values $\varepsilon = 3.5$ are observed in the samples treated during 60 minutes in aqueous solution of glycerine (Fig. 3a). The value ε increases to 2.6 with the increase in the frequency of UST already after 20 minutes of treatment in solution of glycerine. At double action in different media there takes place stabilization of the values of ε within 2.5-2.8 (Fig. 3c).

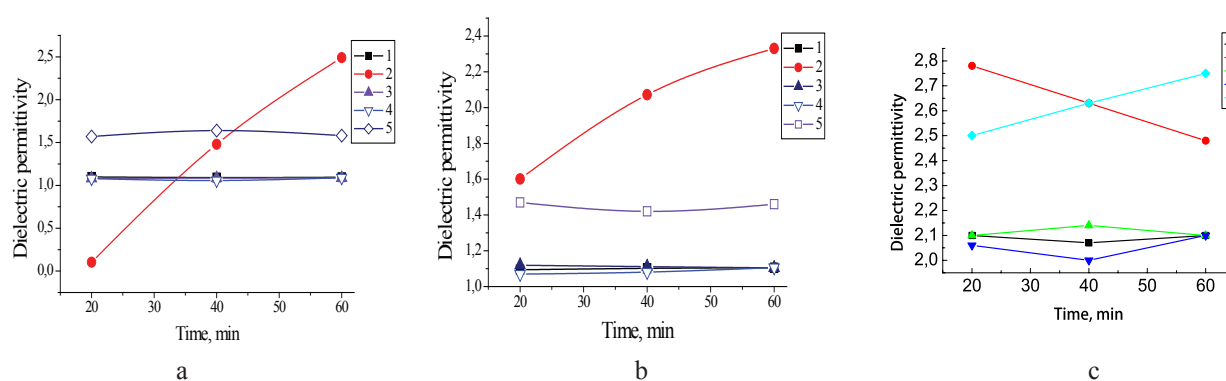


Fig. 3. Dependence of dielectric permittivity on the time of US-treatment of wollastonite in water (1), in aqueous solution of glycerine (2) and polyvinyl alcohol (3), salicylic (4) and silicic acid (5) at 40 (a) 100 kHz (b) and double US-action of 40 and 100 kHz (c).

Figure 3 presents the results of relative dielectric permittivity obtained as the ratio of the capacitor capacitance with dielectric (CD) to the capacitance of an empty capacitor (CO) measured using “Digital multimetr DM-6243L” at the frequency of measurement 800 Hz. The change in the value of charging-discharging current in the circuit leads to the change in the frequency of generation of an electric signal in the measuring circuit. Depending on the structure of the material, the change of current in the circuit leads to the change of polarization processes in the material, this being manifested in the value of the measured dielectric characteristics of the substance. The higher the current in the charge-discharge circuit and the less the dielectric permittivity of the substance being measured, the higher is the frequency of generation of electric signal of the given circuit.

Thus, the results of dielectric permittivity measurements vividly indicate the fact of structural changes taking place during US-treatment on the surface of wollastonite particles. The obtained dependencies on the time of treatment and the pres-

ence of a modifier make it possible to characterize the direction in the change of the material state and its stabilization under concrete conditions of US-treatment.

As follows from the above presented results, depending on the conditions of UST (frequency, time), the character and level of structural changes somewhat differ, this effecting the thermokinetic characteristics of the combustion process. Intensification of the combustion process, when introducing wollastonite into the charge mixture after US-treatment in water, can be related to both the increase of defects of the powder particles and hydration of the surface layer of particles. As was shown earlier, when using mechanochemical treatment for modification of different minerals [12, 13], modification of wollastonite with compounds containing bound water contributes to the increase of thermokinetic characteristics of combustion.

The changes in the value of maximum combustion temperature are most significant (Fig. 4). The figure presents the dependencies of maximum temperature of combustion on conditions of UST of

wollastonite. The maximum combustion temperature was about 1600 °C for the samples containing wollastonite after UST during 40 minutes under simultaneous US-action of 40 and 100 kHz.

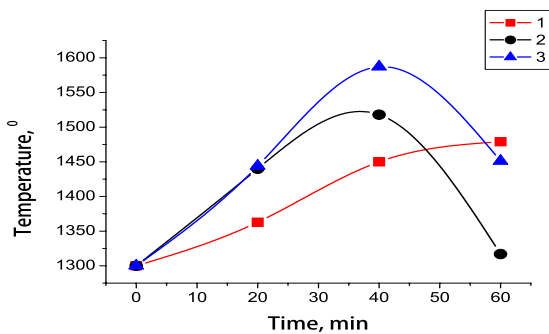


Fig. 4. Dependency of maximum combustion temperature of SHS-samples with 10% wollastonite after US-treatment in water on time at the frequency of 40 kHz (1), 100 kHz (2) and simultaneous action of 40 and 100 kHz (3).

When introducing different modifying additives into the medium of US-treatment of wollastonite, the tendency to intensification of the combustion process is preserved, only parameters of SH-synthesis process somewhat change (Fig. 5). For the samples modified during UST in the presence of polyvinyl alcohol, combustion proceeds more intensively (the rate exceeds 120 degrees/sec) than with wol-

lastonite modified during UST with glycerine when the combustion rate changes from 50 to 90 degrees/sec. However, the combustion temperature reaches 1600 °C. Combustion proceeds most actively on the samples with wollastonite treated by ultrasound at double action of frequencies 40 and 100 kHz.

Dependencies in the change of maximum combustion temperature of the samples with wollastonite modified under different conditions of UST show that after treatment in alcohol solutions, dependencies $T_{\max} = f(\tau)$ have an extremal character. At the frequency of 100 kHz during 20-40 minutes on the surface of particles there forms a structure which contributes to the rise of combustion temperature up to 1500-1600 °C. The structure, which is formed on the surface of wollastonite particles during UST in an acid medium, contributes to a more stable combustion with maximum temperatures of 1550-1600 °C.

Thus, the investigation results of the effect of wollastonite modified during US-treatment on the combustion process showed that its addition considerably activates the combustion process. This is a vivid index of the change in the structure and state of the material under the action of UST. The degree of the material activity is determined by the conditions of US-treatment, namely, it depends on the frequency and time of action. For different media, optimum conditions differ in the frequency and time of treatment. Judging by thermokinetic parameters of combustion, the most effective is the time of 40 minutes at double action of UST of 40 and 100 kHz.

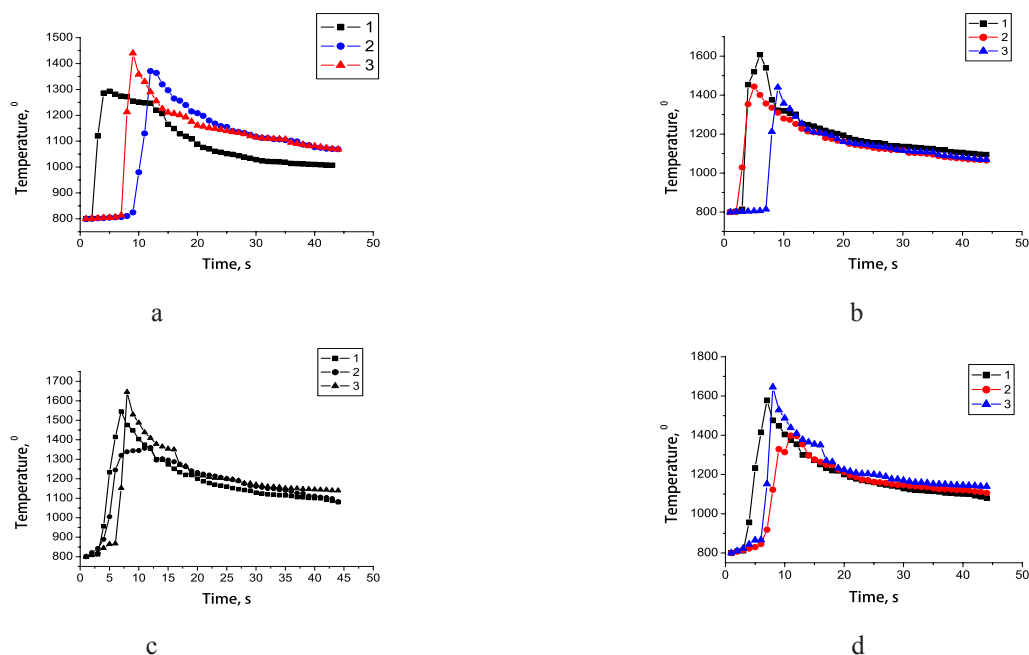


Fig. 5. Combustion thermograms of the system $[\text{SiO}_2+37.5\%\text{Al}]$ with wollastonite additives after US-treatment in 10% aqueous solution of $\text{C}_3\text{H}_5(\text{OH})_3$ (a), $(\text{C}_2\text{H}_3\text{OH})_n$ (b), $\text{C}_9\text{H}_8\text{O}_4$ (c), $\text{H}_2\text{SiO}_3n(\text{H}_2\text{O})$ (d) during 60 minutes at frequencies 40 kHz (1), 100 kHz (2) and simultaneous US-action of 40 and 100 kHz (3).

One of the indexes of quality of the synthesized samples is ultimate strength. If for the sample obtained after SHS of the system ($\text{SiO}_2 + 37.5\% \text{ Al}$) with quartz activated during 10 minutes in a planetary-centrifugal mill, the strength makes up 33

MPa, its values for SHS-samples obtained with wollastonite modified under different conditions of UST significantly change depending on the conditions of US- modification. The results of measurement are presented in Table 2.

Table 2
Strength of SHS- samples of ($\text{SiO}_2 + 37.5\% \text{ Al}$) with 10 % wollastonite modified in different liquid media under different conditions of UST depending on the frequency and time

Conditions of US- treatment		Strength of SHS-samples, MPa				
		Medium of US-treatment of wollastonite				
Frequency f, kHz	Time, min	H ₂ O	H ₂ O+5% C ₃ H ₅ (OH) ₃	H ₂ O+5% H ₂ SiO ₃ nH ₂ O	H ₂ O+5% (C ₂ H ₃ OH) _n	H ₂ O+5% C ₉ H ₈ O ₄
40	20	41.8	50.16	25.08	20.9	62.70
	40	71.06	39.71	21.62	22.99	62.70
	60	62.70	41.80	18.81	35.53	52.25
100	20	33.44	58.52	10.45	14.63	73.15
	40	31.35	12.54	16.75	33.44	62.70
	60	37.62	14.63	45.98	31.35	52.25
100+40	20	41.80	18.81	52.25	39.71	73.15
	40	73.15	73.15	31.35	41.8	62.70
	60	29.26	20.90	27.17	20.9	62.70

The samples containing wollastonite after US-treatment in water and aqueous solution of glycerine showed the highest strength (73.15 MPa). The samples with wollastonite modified with silicic acid showed the lowest strength. Treatment of wollastonite in solution of salicylic acid contributes to stable increase in the strength of SHS-samples (from 52 to 73 MPa) independent of the conditions of US- treatment, this vividly following from the data presented in Table 2. The samples synthesized with wollastonite modified during US-treatment in solution of salicylic acid at simultaneous action with frequencies 40 and 100 kHz are characterized by high stable values of strength (62-73 MPa) (Fig. 6).

Table 3 presents the results of X-ray phase analysis of synthesized samples under the conditions of US- treatment of wollastonite introduced into the charge which provide the most significant changes in the strength of the synthesized sample.

In all cases, introduction of wollastonite after US-treatment into the charge contributes to a more complete conversion of the initial components in the combustion process of the samples. The greatest amount of corundum and silicon is formed, when using wollastonite treated in water, aqueous solution of polyvinyl alcohol and salicylic acid. The presence of iron containing phases in the reaction products is due to the presence of iron impurities in the used quartz material. Of certain interest are the results on

the phase analysis of SHS samples with wollastonite modified in the presence of glycerine and silicic acid. In these samples, compounds $\text{C}_9\text{H}_{10}\text{Cl}_2\text{N}_2\text{O}$ and $\text{Na}(\text{AlSi}_3\text{O}_8)$ are formed, the presence of which can be considered to be conditioned by formation, on the surface of particles during US-treatment, of compounds in which participates carbon in the composition of glycerine, chlorine and sodium which are present in water (the experiments were carried out with tap water) and nitrogen the presence of which in water and consequently, in cavitation bubbles is quite real.

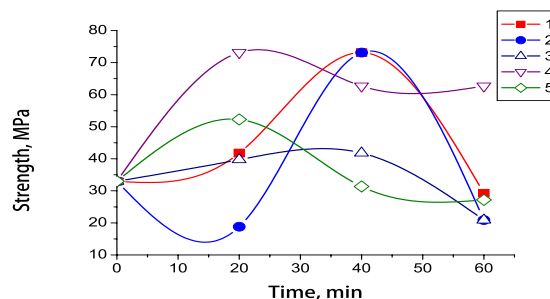


Fig 6. The change in the strength of SHS- samples with wollastonite modified depending on the time of UST in water (1), aqueous solution of glycerine (2), polyvinyl alcohol (3) salicylic acid (4) and silicic acid (5). UST at simultaneous action with 40 and 100 kHz.

Table 3

The phase composition of the reaction products of $\text{SiO}_2 + \text{Al}$ samples with wollastonite depending on the medium of US- treatment during 40 minutes at simultaneous action of 40 and 100 kHz

Medium of UST	The phase composition, %								
	Phases								
	Al_2O_3	Si	FeSi_2	FeAl_3Si_2	Fe_2O_3	$\text{C}_9\text{H}_{10}\text{Cl}_2\text{N}_2\text{O}$	$\text{Na}(\text{AlSi}_3\text{O}_8)$	SiO_2	Al
	48.4	12.9	-	1.8				24.7	12.2
H_2O	67.3	14.7	1.0	1.6	0.8			9.2	5.4
$\text{H}_2\text{O} + 10\% \text{C}_3\text{H}_5(\text{OH})_3$	46.6	12.3	0.4			4.7	5.6	16.3	14.1
$\text{H}_2\text{O} + 10\% \text{H}_2\text{SiO}_3 \cdot n\text{H}_2\text{O}$	49.8	15.1	6.5	1.6			6.2	8.5	12.2
$\text{H}_2\text{O} + 10\% (\text{C}_2\text{H}_5\text{OH})_n$	65.0	14.2	1.3	4.1	1.9			7.8	5.7
$\text{H}_2\text{O} + 10\% \text{C}_9\text{H}_8\text{O}_4$	72.6	21.0	0.6		0.6			2.9	2.3

Another important factor is also the fact that such values of the permittivity of the material does not exceed $\epsilon = 2.5$, which is also characterized by its high insulating properties.

Thus, US-treatment of wollastonite powder being introduced into the composition of charge mixture is an effective method of purposeful changing of both mechanical and physical properties of SHS-material. In particular, the possibility of improving electro- insulation properties of SHS-material the main phase of which is corundum is shown.

Conclusions

The results of investigations on the effect of ultrasonic treatment (UST) of wollastonite (brand FW 100) in water and 10% aqueous solutions of different alcohols and acids at 40 and 100 kHz as well as simultaneous action of UST of 40 and 100 kHz showed that dispersity of the powder increases depending on both the time and frequency of US- action. The greatest effect is registered at simultaneous action by frequencies 40 and 100 kHz, i.e. there takes place a synergic effect of simultaneous action of low and high US frequencies when cavitation bubbles appearing at low frequencies get a great acceleration and the action of hydrodynamic flows created by ultrasound vibrations of high frequency.

Electron microscopic investigations showed grinding and erosion of the surface layer of particles or sticking of particles together due to modification of their surface depending on the conditions of UST and the medium of treatment. US- modification in the presence of silicic acid and polyvinyl alcohol in aqueous solution contributes to enlarging of particles. After treatment in aqueous solution of glycerine, film layer formations are formed on the surface. After US- treatment in aqueous solution of salicylic acid, the powder is presented by aggregates of fragments of different dispersity and morphology

combining semitransparent formations and dense particles. The degree of the material activity is determined by the conditions of US-treatment. It is stated that the samples containing wollastonite after UST during 40 minutes at simultaneous action of 40 and 100 kHz show maximum temperature of combustion exceeding 1600 °C.

A more complete conversion of initial components in the combustion process of the samples is stated to take place, when introducing wollastonite into the charge after US-treatment. The greatest amount of corundum and silicon is formed, when using wollastonite treated in water, aqueous solution of polyvinyl alcohol and salicylic acid. Such samples are characterized by high stable values of strength (up to 73 MPa) and indexes of electroinsulation properties.

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