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**Mechanical Activation as Sintering Pre-treatment of Talc for Steatite Ceramics****A. Terzić<sup>1\*)</sup>, Lj. Andrić<sup>2</sup>, J. Stojanović<sup>2</sup>, N. Obradović<sup>3</sup>, M. Kostović<sup>4</sup>**<sup>1</sup>Institute for Materials Testing, Vojvode Mišića Boulevard 43, 11000 Belgrade, Serbia<sup>2</sup>Institute for Technology of Nuclear and other Raw Mineral Materials, Franchet d'Esperay st. 86, 11000 Belgrade, Serbia<sup>3</sup>Institute of Technical Sciences, Serbian Academy of Science and Art, Knez-Mihailova 35, 11000 Belgrade, Serbia<sup>4</sup>Faculty of Mining and Geology, University of Belgrade, Đušina 7, 11000 Belgrade, Serbia**Abstract:**

The effect of vibratory mill induced mechanical activation on the change of the particle size, crystallinity and the phase transformations of the minerals present in the activated material, was studied with the purpose of decreasing of the sintering temperature of talc ( $Mg_3Si_4O_{10}(OH)_2$ ) as raw material which is the basic component of the steatite ceramics. The aims of the conducted investigation were, also, increasing of the reactivity of the comminuted raw material and establishing of the optimal activation period. The properties of the activated talc induced by mechanical force were expressed in form of the grain inertia change which was measured by means of automatic grain counter. Mechanically activated grains are the most convenient mineral form for physical concentration since the energy change of the mill-material system is recorded on them. The effect of dry grinding on the structure, particle size and shape of talc was studied by means of XRD, DTA and SEM/EDS methods. Activation of talc produced an increase of the starting surface area value progressively from 4.5 m<sup>2</sup>/g up to a maximum of 108.5 m<sup>2</sup>/g achieved at 30 min. A subsequent decrease of rate of surface area change and the rate of size reduction were observed following the prolonged grinding. Talc activated in vibratory mill for optimal 30 min showed properties which positively influence the decrease of sintering temperature and the increase of the sintering rate of steatite ceramics.

**Key words:** Energy conversion, Grain potential, Vibratory mill, Sintering, Steatite ceramics.

**1. Introduction**

Steatite is a magnesium silicate composite material which is produced by means of the standard ceramic processing methods and can readily be machined or sintered into a variety of forms. Steatite ceramic is widely used in electronic engineering (electrical insulation, regulator bases, switches and plug parts, sockets bases for halogen lamps, heating

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element holders, etc.) due to its excellent electrical properties, high mechanical resistance, low dielectric loss and high temperature resistance [1-3]. Other than in electronics, the steatite ceramic is used in medicine as material for dental implants due to high hardness and bending resistance, and as material for artificial bone due to good bio-compatibility [4, 5]. The raw materials commonly used in steatite ceramics synthesis are: talc mixture for calcination procedure, montmorillonite as bonding agent, and feldspar or  $\text{BaCO}_3$  as melting agents. Synthesis is normally conducted at approximately  $1400^\circ\text{C}$ . Synthesis product is a crystalline phase of magnesium metasilicate ( $\text{MgSiO}_3$ ) obtained from talc, while melting agent forms a vitreous phase which melts and surrounds the crystalline phase [6, 7].

Talc is a hydrated magnesium silicate  $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$  with the structure consisting of a brucite octahedral sheet bonded to two silica tetrahedral sheets by oxygen atoms [8]. Talc is frequently applied in industry as raw material due to its chemical inertia, softness, high thermal stability and low electrical conductivity [9]. Steatite ceramics, made up of approximately 70 % of magnesium metasilicate and 30 % of vitreous phase, is one of the most important technological applications of talc [10]. The talc used in these ceramics must meet certain technical criteria, the most important one of which is iron content, because it affects the whiteness and the refractory properties of ceramics [11]. A problem affecting steatite ceramics is the formation of vitreous silica during talc calcination, since its presence negatively alters the dielectric properties of ceramics [12].

Mechanical activation is a procedure which is frequently applied as a means of improvement of characteristics of comminuted ore grain mixtures used as component in the design of ceramic materials [13, 14]. The activation of a solid substance is accomplished by the fine and ultra-fine processing of the material in the specially designed high-energy mills [15]. Activation does not only effect the change of the particle size, it is a complex physical-chemical process which induces the increase of potential energy, chemical activity and surface reactivity of the system [16]. The main principle lying behind activating technology relies on changed energy properties of processed solid materials, i.e. on gravitational and electromagnetic properties. These properties are changed due to a changed energy condition of the processed grains, induced by the activity of mechanical energy [17, 18]. Namely, activation process is the system, which comprises the fields of the active forces, devices and mineral grains, in which the centrifugal acceleration may exceed gravitational acceleration by 100 times. In the centrifugal field, mineral grains are brought together by a strong force, whereby the gravitational force acting within a narrow space around particles exceeds weight of the grains by several tenths times. Such a loading of material results in elastic and plastic modifications, changing of the shape, volume and surface density of the grains. The energy properties induced by mechanical force are best expressed as the change of the grain inertia [19]. The characteristics of the activated talc and the possibility of the grain inertia measurement by automatic grain counter were investigated and presented in this paper. The reactivity of the material is improved since the material structural parameters are changing due to the excess of free energy and volume [20, 21]. The increase of material reactivity can be efficaciously used in rationalization of process and making the basic technologies cheaper, first and foremost reducing the sintering temperature and increasing the rate of sintering.

## 2. Experimental procedure

The raw talc from "Bela Stena" deposit, Serbia was used in this study. Comminuting of talc ore was performed by crushing and grinding up to the particle size that could be used as input for the activation. The result of the chemical analysis of the talc is given in Tab. I.

**Tab. I** Chemical composition of initial talc sample.

Oxide	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	MgO	Fe <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	Ti <sub>2</sub> O	LoI
%	1.92	39.25	4.69	29.25	3.90	0.02	0.01	0.01	20.95

The obtained bulk density of the talc sample, according to Standard SRPS EN 725-8:2010, was 1.62 g/cm<sup>3</sup>. The grain mixture that underwent further investigation was obtained after treatment which included reduction of the ore particle size. Primary crushing of the raw talc sample was conducted by jaw crusher, with 10 mm output opening, working in a closed circle with screen. Afterwards, primary crushed sample was subjected to crushing in the roll crusher with 5 mm output opening. Grinding of the secondary crushed sample was performed in the ceramic-lined ball mill with ceramic balls as grinding media.

The talc sample was activated by means of the laboratory mechano-activator – vibratory disc mill RS 200 (Retsch, Düsseldorf, Germany). The RS 200 mill comminutes by impact and by friction. Batch size of the mill is 35 – 150 ml. Material of grinding tools is hardened steel. The vibratory mill is activator in which energy is transferred directly onto activating elements. In order to explain theoretical principles of activating operation it is necessary to divide influential factors in two groups: working and variable factors. Total volume of space available for activation, circumferential velocity, and rocking amplitude are working parameters; and total ring mass, number and diameter of the activating bodies, and structural characteristics of the material are variable parameters [22]. Vibration, amplitude, impact and contact surface between grinding bodies and processed material increase with kinetic energy generating in activating bodies. When kinetic and potential energy become equal, the following equations are obtained [22]:

$$\frac{m \cdot v_v^2}{2} = H \cdot G = H \cdot m \cdot g \Rightarrow H = \frac{v_v}{2 \cdot g} = \frac{(r\omega \cdot \cos \beta)^2}{2 \cdot g} \cdot m \quad (1)$$

Where: *m* is activating body mass, kg; *g* is constant of gravity, m<sup>2</sup>/s; and *H* is the height of elevation and subsequent free fall of the activating body, m.

The height is not only dependent on centrifugal acceleration ( $a = r \cdot \omega^2$ ), it is also dependent on the value of amplitude (*r*). By knowing the value of the centrifugal acceleration (*a*), and the number of revolutions (*n*) of the engine, the values of amplitude (*r*) and frequency (*f*) can be calculated from following equations:

$$r = \frac{a}{\omega^2} \quad \text{and} \quad f = \frac{1}{T} = \frac{\omega}{2\pi} \quad (2)$$

The activation of talc experiment was based on grinding kinetic model which means that achieved results can be described by Rosin-Rammler diagram and equations [22]. The characteristics of activated product are predominantly a function of the mechano-activator type. The strain intensity and number of impulses are the basic variable parameters which influence activation kinetics and consequently the activation product. Cumulative characteristics of the size of activated material can be described by functional dependency between the mean diameter, cumulative oversize - *R*, and undersize - *D* [22]:

$$R = 100e^{-\left(\frac{d}{d_{50}}\right)^n} \quad (3)$$

Where: *R* is cumulative oversize; %; *e* is basis of natural logarithm (*e* = 2,718); *d* is sieve opening; mm; *d*<sub>50</sub> is mean grain diameter; mm.

By performing a double logarithm of the Rosin-Rammler function (Eq. (3)), a new equation which represents straight line equation with the direction coefficient – *n* is obtained:

$$n = \frac{\log \log \frac{100}{R_1} - \log \log \frac{100}{R_2}}{\log d_1 - \log d_2} \quad (4)$$

Parameter  $d_{95}$  (sieve opening through which passes 95 % of the activated product) can be also calculated by multiplying Eq. (3) with the natural logarithm [22]:

$$d_{95} = e^{\left(\frac{n \ln d_{50} + \ln \ln 100 - \ln \ln R}{n}\right)} \quad (5)$$

Theoretical specific surface area can be calculated from the mean diameter as [22]:

$$S_t = \frac{6.39}{\rho \cdot d} \cdot e^{\frac{1.795}{n^2}} \quad (6)$$

Where:  $S_t$  is theoretical specific surface area,  $m^2/kg$ ;  $d_{50}$  and  $n$  are Rosin-Rammler equation parameters;  $e$  is basis of natural logarithm ( $e = 2.718$ ); and  $\rho$  is density;  $kg/m^3$

Automatic grain counter provides response in the form of the voltage pulse, generated as the grain passes through the opening, its value directly proportional to the grain volume. The resistance of electrolyte including the grain is equal to resistance of two parallelly connected resistors, form which follows the mathematical relation between the counter response and grain size [21]:

$$\delta R = \frac{1}{\frac{A-a}{\rho_f \cdot \delta_\lambda} + \frac{a}{\rho_s \cdot \delta_\lambda}} \quad (7)$$

Where:  $\rho_s$  and  $\rho_f$  denote resistances of grain and fluid, respectively;  $A$  is the cross section of the opening;  $a$  is the cross section of the grain;  $\delta_l$  is thickness of the element or the segment.

External resistance in circle is high enough to analyze small change of the opening resistance caused by the grain presence,  $\Delta R$ , i.e. its influence on the current density,  $I$ . Generated voltage pulse is:  $I \cdot \Delta R$ . It was experimentally determined that response is independent on the grain resistance [21]. Namely, the response is independent on the grain resistance due to oxide films present on the surface and ionic inertia of the Helmholtz double electric layer and solvent molecules adhered on the grains surface, whereby their electric resistance becomes infinite [21]. The response is not in proportion with grain volume, instead it is modified due to the presence of  $a/A$  ratio:

$$\delta(\Delta R) = \frac{\frac{\rho_f \cdot a \cdot \delta_l}{A_2}}{1 - \frac{a}{A}} \quad (8)$$

The described measuring method is based on determination of grain coarseness using the data obtained for counted grains and the assumption that generated voltage is proportional to the grain volume. The custom practice in techniques is that one physical value is representing the change of another; thus the automatic grain counter operating on this assumption holds that generated voltage is representing the change of the grain inertia. In other words, the value recorded by a solid matter and read by the automatic grain counter, has no influence on the instrument response. As it comes out, voltage is the measure of the grain inertia, not its volume. Inertia of spherical grain in its gravity center can be calculated as:

$$J = m \cdot r^2 \quad (9)$$

Where:  $m$  is mass of the grain,  $kg$ ; and  $r$  is the grain radius.

Specific surface area for the sphere, SSA, is:

$$SSA = \frac{d_{50}^2 \cdot \pi}{m} \quad (10)$$

The hypothesis that automatic grain counter actually measures change of inertia, i.e. change of the mechanical energy state, can be represented as:

$$J = \frac{3 \cdot V \cdot d_{50}}{2 \cdot SSA}, \text{ kgm}^2 \quad (11)$$

Where:  $d_{50}$  is mean grain size, m; and V is volume,  $\text{m}^3$ .

The chemical analysis of the talc was performed by atomic emission spectroscopy technique: PinAAcle 900 Atomic Absorption Spectrometer (Perkin Elmer, Waltham Massachusetts, USA). The specific surface area of initial talc sample was obtained by Brunauer–Emmett–Teller, BET method. The grain fraction content of talc was analyzed by cyclo-sizer diffraction particle size analysis (Warman International LTD, Australia). Mineralogical changes in talc were analyzed by X-ray powder diffraction (Philips PW-1710 diffractometer). The microstructure of the samples was characterized by scanning electron microscopy method using a JSM-6610LV apparatus (Tokyo, Japan) equipped with energy dispersive X-ray spectroscopy (EDS). For SEM analysis, the samples were coated with Au films to improve conductivity prior to imaging.

### 3. Results and discussion

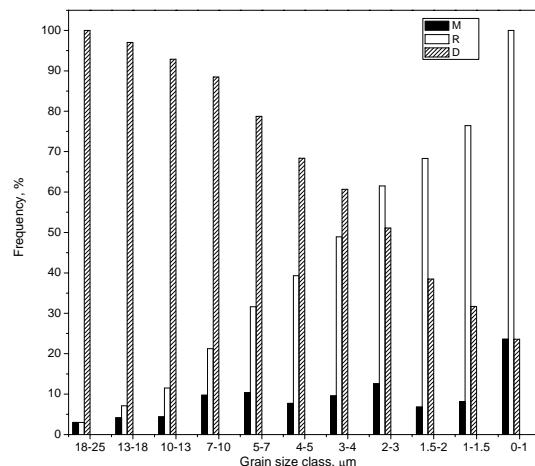
Prepared talc grain mixture underwent activation procedure in the vibratory mill following the previously given procedure description. The samples were analyzed after a sequence of activation periods: 5, 10, 15, 20, 30, 40, 50, 60 and 90 minutes. The theoretical results calculated from functions (Eq. 1-6) were compared with results obtained by means of automatic grain counter (Eq. 7-11) and the survey of the analysis data for all activated samples is given in Tab. II.

**Tab. II** Survey of the analysis data for samples obtained after various activation periods.

Activatin period, min	Automatic grain counter						Rosin-Rammler function				
	SSA, $\text{m}^2/\text{g}$	$V, \text{m}^3$	$d_{50}, \text{mm}$	Grains numb.	$V/SSA, \text{m} \cdot \text{g}$	$J, \text{kg} \cdot \text{m}^2$	$d_{50}, \mu\text{m}$	$d_{95}, \text{mm}$	$S_t, \text{m}^2/\text{kg}$	$W_e, \text{kWh/t}$	Agglom. tendency
0	4.5	-	28.65	-	-	-	28.65	59.18	4.5	-	-
5	11.2	$5.999 \cdot 10^{-3}$	7.35	9235	$1.87 \cdot 10^{-4}$	$2.81 \cdot 10^{-7}$	7.40	11.58	11.5	153.2	no
10	19.5	$5.585 \cdot 10^{-3}$	6.21	10036	$3.49 \cdot 10^{-4}$	$3.01 \cdot 10^{-7}$	7.10	11.02	30.6	215.22	no
15	38.2	$4.789 \cdot 10^{-3}$	5.74	10125	$7.85 \cdot 10^{-4}$	$1.07 \cdot 10^{-7}$	5.35	10.35	40.2	259.96	no
20	57.5	$3.752 \cdot 10^{-3}$	4.38	11322	$15.2 \cdot 10^{-4}$	$0.42 \cdot 10^{-7}$	4.26	9.58	59.5	264.25	no
30	108.5	$2.564 \cdot 10^{-3}$	2.51	12298	$42.7 \cdot 10^{-4}$	$0.09 \cdot 10^{-7}$	2.56	8.69	108.5	280.57	no
40	108.3	$2.005 \cdot 10^{-3}$	2.48	12999	$54.3 \cdot 10^{-4}$	$0.09 \cdot 10^{-7}$	2.56	7.15	108.1	300.56	no
50	108.0	$2.005 \cdot 10^{-3}$	2.43	13025	$53.9 \cdot 10^{-4}$	$0.09 \cdot 10^{-7}$	2.50	7.10	108.0	328.21	no
60	107.9	$2.005 \cdot 10^{-3}$	2.39	13058	$53.9 \cdot 10^{-4}$	$0.09 \cdot 10^{-7}$	2.45	7.10	107.5	354.89	weak
90	107.5	$2.000 \cdot 10^{-3}$	2.35	13099	$53.8 \cdot 10^{-4}$	$0.09 \cdot 10^{-7}$	2.40	7.05	107.0	395.45	strong

The specific surface area of talc increased from  $4.5 \text{ m}^2/\text{g}$  to  $108.5 \text{ m}^2/\text{g}$  as a result of activation treatment. The maximal value of the specific surface area is obtained for the talc sample activated for 30 min. During the first 30 min of the activation period specific surface area was rapidly increasing, and subsequently from 30-90 min the rate slowed down. The relative increases in specific surface area were significant, particularly during first 30 min of activation, due to high content of impurities [23]. It is noticed that grain inertia value is also in function of activation period, the values of inertia being lower when the activation period was longer. The decrease of the value of inertia consolidated around 30 min. The grain inertia change can be explained by the theory that extension of the activation period influences increasing of the tribo-mechanical interaction between the grains, i.e. it supports the loss of electrons and in that way the grain mass is reduced [24]. In this case, the grain mass is assessed indirectly, by measuring grain inertia by means of the automatic grain counter (AGC). Namely, the variations in mean grain diameter, specific surface area, total volume and number of grains are variables which determine one physical parameter: the grain inertia momentum. The grain inertia momentum reflects expected regularity in variation of mechanical properties of grains after conducted activation, which is the agreement with the model of the grain which lost its electrons due to tribo-mechanical effect and remained positively charged with a smaller mass [25-26]. Talc contains three groups of mineral oxides. The first group includes the minerals with metal element -  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{CaO}$ ,  $\text{MgO}$ ,  $\text{Na}_2\text{O}$  and  $\text{K}_2\text{O}$ . Participation of metal elements in the sample amounts to 30 %. The second group of is represented by  $\text{SiO}_2$ , with Si being a metalloid. Participation of Si oxides is 40 %. The third group of minerals includes nonmetallic elements (30 %). The hypothesis is that the grain which lost its electrons due to tribo-mechanical effect either remains positively charged with reduced mass, or its electrons remain built in the structure of the crystal lattice composed of nonmetallic crystallites and its mass increases [27]. In the activated talc sample, metals remained positively charged due to electron losses, while nonmetals were negatively charged due to acceptance of electrons. Activated talc was analyzed using AGC to prove that mass of the grains is more often reduced (inertia decrease) then increased (inertia increase), relative to participation of minerals "divided" in the activated material. Thus, the tendency of the grain inertia decrease, accompanied by mass reduction is depending on mechanical activation period as it was shown in Tab. II.

In Fig. 1., the result of the grain-size analysis, i.e. histogram of mass portion, M; oversize, R; and undersize, D, of the talc activated for 30 min is given.

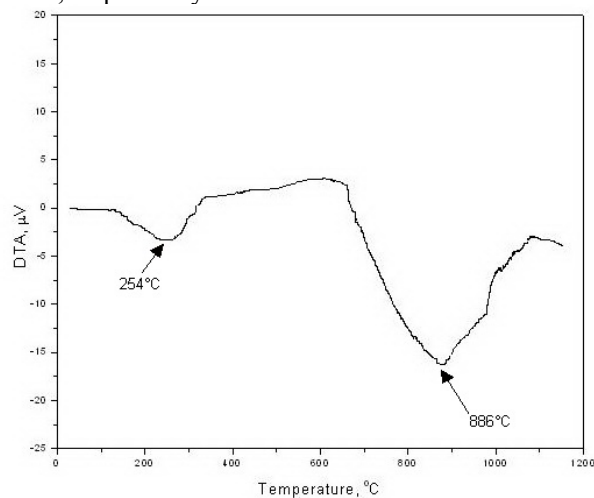


**Fig. 1.** The granulometry of 30 min activated talc sample.

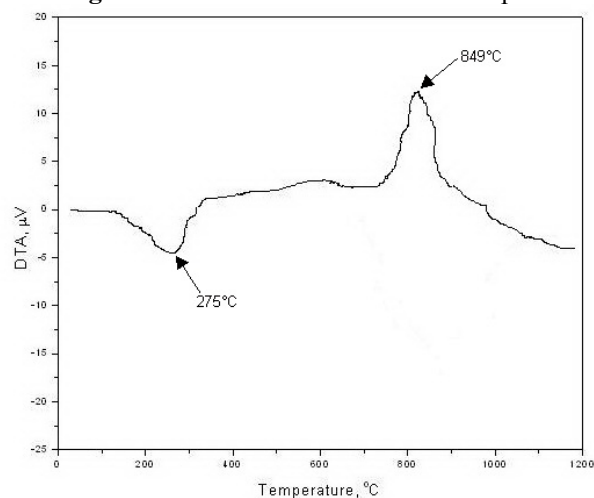
In order to explain the influence of activation on the structure and characteristics of talc, a number of specific analyses were conducted before and after activation procedure:

differential thermal analysis (DTA); X-ray powder diffraction analysis (XRD) and scanning electron microscopy (SEM/EDS).

Processes taking place in the non-activated and activated system during thermal treatment from ambient temperature towards 1100°C, under the heating rate of 10°C/min, were identified by means of the DTA. The DTA curves of original and ground talc samples are given in Fig 2. and 3., respectively.



**Fig 2.** DTA curve of the initial talc sample.



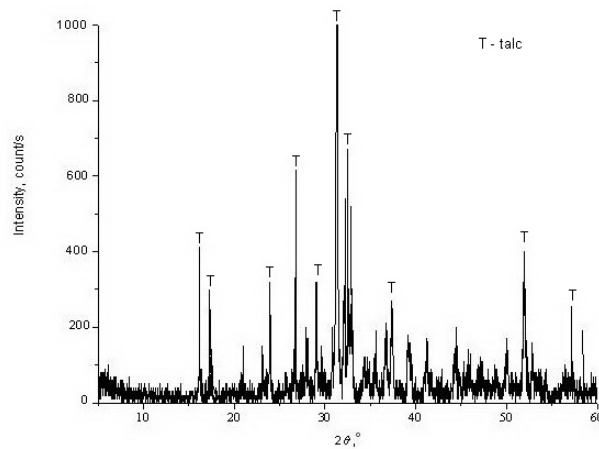
**Fig 3.** DTA curve of the talc sample activated for 30 min in vibratory mill.

In the non-activated talc system given in Fig. 2., all the major processes taking place during heating correspond to the endothermic peaks appearing on the DTA curve. The first major peak below 300°C corresponds to the loss of water adsorbed on the particles surfaces, while the smaller effects appearing above 300°C correspond to the subsequent loss of OH groups [9, 28]. The endothermal effect appearing at approximately 900°C corresponds to the loss of structural water which is accompanied by the enstatite ( $\text{MgSiO}_3$ ) and silica formation [9, 28].

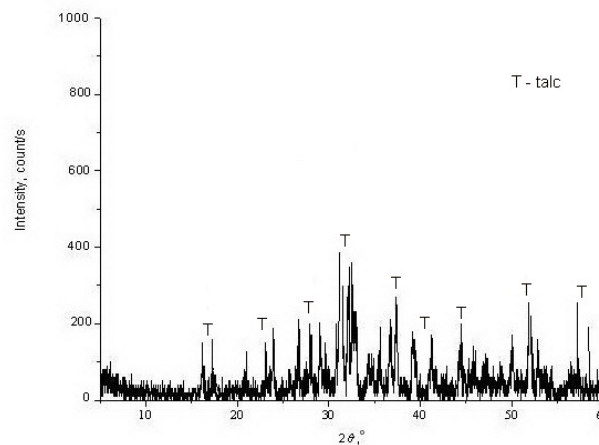
The comparison of DTA curves of the original and ground talc sample pointed out to a slight shift towards higher temperature in the main endothermal DTA peak of ground sample which is associated to talc dehydroxylation. This fact can be related to the decrease of bond energy of OH groups linked to Mg ions because grinding stresses the crystal structure

and causes that the bond energy of these groups tends to decrease. Thusly, the diminution of particles sizes and evolution of surface area of the ground sample are both associated with the weekly bound OH groups on the broken edges of the ground talc particles [29]. As it can be seen from Fig. 3., the activation altered the DTA curve of talc, inducing the formation of high temperature phases [30]. Namely, an exothermal effect detected above 800°C marked the crystallization of non-crystalline phases formed by mechano-activation of the talc sample, into orthorhombic enstatite [28]. Besides enstatite, amorphous silica is formed during this process [31]. However, activation enhanced the quantity of the enstatite which is mineral with high melting point and by such prevented negative effect of vitreous silica at elevated temperature. Namely, the enstatite is produced by the reactions between magnesium chloride, oxygen and vitreous silica, the reaction causing the removal of vitreous silica, which is the main impurity that affects the dielectric properties of steatite ceramics. Mechanical activation can be an efficient method for obtaining higher amounts of protoenstatite, which is one of the main components of steatite ceramics, at relatively low temperatures - 800°C. Formation of high-temperature phases in activated material positively influences the rate of sintering making the process faster.

Mineralogical composition and changes in crystal structure of the initial talc sample and the sample activated for 30 min are given as XRD diffractograms in Fig. 4. and 5.



**Fig. 4.** XRD diffractogram of initial talc sample.



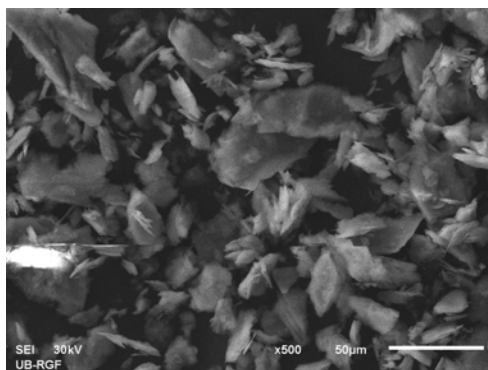
**Fig. 5.** XRD diffractogram the talc sample activated for 30 min.



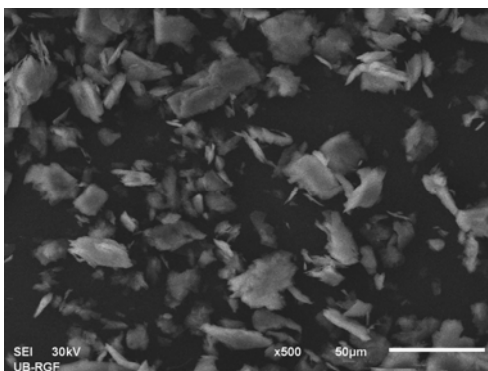
Major identified crystalline phase present in both analyzed samples is the talc mineral. i.e. the X-ray analysis showed presence of 98% of pure talc accompanied only by negligible amounts of chlorite and quartz. The activation procedure did not promote formation of new mineral phases. However, the crystallinity of the activated talc was noticeably lower than that of initial sample which can be attributed to the effect of activation. Certain changes in the crystalline structure appeared within 5 min of the activation; however the more significant results concerning reduction of the sample crystallinity were reserved for longer activation periods. The crystallinity decreasing its rate after 30 min of activation; i.e. mechanical reduction of the original particles appears to have reached a limit at 30 min grinding time. Longer grinding times might produce contra effect - an increase in particle size.

It can be concluded that activation influences the crystalline structure of the talc, i.e. level of crystallinity decreased with increasing activation time, and that activation made the structure disordered and generated crystal lattice defects and other meta-stable forms. The activation treatment might promote the amorphization of treated material, noticeable change of the micro-structure, size and shape of particles, etc.

The previously noticed changes occurring with the microstructure of activated talc were further investigated by SEM method. In Fig. 6. and 7. the SEM microphotographs of talc samples before activation and after 30 min of activation are given, followed by accompanying EDS analysis (Fig. 8.).



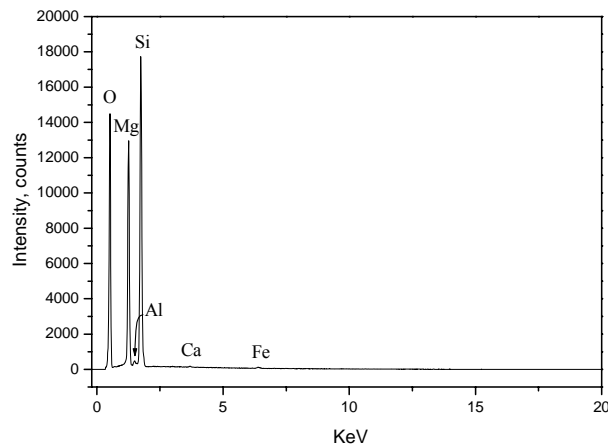
**Fig. 6.** SEM microphotograph of initial talc sample.



**Fig. 7.** SEM microphotograph of activated talc sample (30 min).

The shape of original talc particles (Fig. 6.) is normally pseudo ellipsoidal to slightly hexagonal, and elongated [32-34]. Dimensions of the non-activated talc particles vary in a significant range. The SEM microphotograph of talc sample after activation (Fig. 7.) shows that the talc particles gained more angular and needle-like shapes. The activated talc particles possess a semi-layered structure and their size is significantly reduced and more uniform. The

SEM analysis of activated talc did not reveal particle size increase or grain agglomeration, therefore it can be concluded that 30 min can be considered as optimal grinding time which does not produce any contra effects. Furthermore, in the case of talc processing, mechanical activation can be considered as a post-treatment in processing of ore mixtures because hydrometallurgical concentration procedure is not necessary. Performed morphological and semi-quantitative analysis indicated that sample corresponds to monoclinic silicate mineral – talc with following chemical formula:  $Mg_3Si_4O_{10}(OH)_2$ .



**Fig. 8.** Semi-quantitative EDS analysis of talc sample activated for 30 min.

The steatite ceramics is based on the formation of a crystalline phase of magnesium metasilicate from the talc mineral. Basic components for steatite ceramics are talc agglomerated with clay and barium carbonate. If barium carbonate is used as the flux for generating vitreous phase the dielectric properties of steatite ceramics usually appear optimal. The sintering of the steatite ceramic normally takes place at 1380–1410°C in the presence of a liquid phase. The microstructure after cooling is composed of crystalline magnesium metasilicate grains surrounded by a vitreous matrix. Among the three possible magnesium metasilicate polymorphs - protoenstatite, ortoenstatite and clinoenstatite, only the first one is thermodynamically stable at high temperature. Mechanical activation conducted in this investigation enhanced the production of thermally-stable form of enstatite in talc and thusly promoted decreasing of the sintering temperature and increasing of the sintering rate. The activation process leads to a decrease of the average grain size of the crystalline phase. This is also expected to improve the aging behavior of the material since large grains seem to show a strong trend to transform from protoenstatite to clinoenstatite. Furthermore, understanding of the microstructural development is a critical factor in the design of steatite cearmics which have to satisfy the increasing demands regarding performance imposed by new applications.

#### 4. Conclusions

During talc activation in vibratory mill a significant decrease of grain size was noticed; the reduction being effected by the strain increase due to the friction, compression and impact forces. The talc showed no tendency for creating agglomerations up to long activation periods (60 min). Activation influenced talc microstructural properties, energy state, reactivity, and crystallinity. Consequently it changes and enhances performing properties of steatite ceramic in which activated talc is applied as basic raw material.

Theoretical parameters of the optimal 30 min talc grinding are obtained in accordance with activation kinetics and Rosin-Ramler mathematical model:  $d_{50} = 2.56 \mu\text{m}$ ;  $d_{95} = 7.15 \mu\text{m}$ ;

specific surface area 108.5 m<sup>2</sup>/kg. Approximately same parameters are obtained according to the operating hypothesis of the automatic grain counter:  $d_{50} = 2.51 \mu\text{m}$ ; specific surface 108.5 m<sup>2</sup>/kg. Thus, the operating hypothesis of AGC is confirmed by Rosin-Ramler mathematical model. The tendency of the grain inertia decrease, accompanied by mass reduction, is depending on mechanical activation. According to model presented, grains lost electrons due to tribo-mechanical effect remaining positively charged, with reduced mass.

X-ray analyses of talc confirmed that activation contributes to the decreasing of crystallinity. The activation promotes amorphization of mineral, microstructural changes, and changes in size and shape of minerals particles. DTA highlighted the fact that activation enhanced the quantity of the enstatite which is mineral with high melting point and by such prevented negative effect of vitreous silica at elevated temperature. Formation of high-temperature phases in activated material positively influences the rate of sintering making the process faster. Reduction of the original particles of talc appeared to have reached a limit at approximately 30 min grinding time and longer activation times might produce an increase in particle size or agglomeration of the particles. The SEM analysis of the talc revealed significant particle size decreasing, and highlighted that there was no appearance of grains agglomeration during 30 min grinding time. Since the grain diameter did not significantly change during activation from 30 to 90 min, from energetic point of view the period of 30 min can be adopted as optimal activation period considering the energy consumption.

The present work was focused on the activation of talc as a ceramic raw material in order to understand and control the microstructure development. The next step will be production and investigation of sintering of steatite ceramics based on activated talk which satisfies increasing demands regarding performance imposed by new industrial applications.

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**Садржај:** Проучаван је утицај механичке активације помоћу вибрационог млина на промену величине зрна, кристаличност и фазну трансформацију минерала присутних у активираним материјалу, а у циљу снижења температуре синтеровања талка ( $Mg_3Si_4O_{10}(OH)_2$ ) као компоненте која се користи за добијање стеатитне керамике. Остали циљеви истраживања били су повећање реактивности уситњеног материјала и утврђивање оптималног периода механо-активације. Својства механички активираним талка уз помоћ механичке силе су изражена у форми промене инерције зрна која је мерена помоћу аутоматског бројача зрна. Механички активирани зрна су најповољнији минерални облик физичке концентрације јер се на њима бележи промена енергије у систему млин-материјал. Утицај сувох млевења на структуру, величину зрна и облик зрна талка проучаван је помоћу XRD, DTA и SEM/EDS метода. Механичка активација талка узроковала је прогресивно повећање почетне специфичне површине од  $4.5 \text{ m}^2/\text{g}$  до максимума од  $108.5 \text{ m}^2/\text{g}$  који је постигнут након 30 мин активације. Смањење брзине промене специфичне површине и смањење брзине редукције величине зрна су примећени у току продуженог млевења. Талк који је активирани у вибрационом млину оптималних 30 мин показује својства која могу позитивно да утичу на снижавање температуре синтеровања и повећање брзине синтеровања стеатитне керамике.

**Кључне речи:** Талк, енергетска промена, потенцијал зрна, вибрациони млин, Механичка активација, синтеровање, стеатитна керамика.

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