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obnovljive izvore
električne energije
pri SMEITS-u**

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FOREWORD

Science, technology and industry accelerated development leads to improvements of human life, but also creates new risky situations. Humanity faces unprecedented risks. Global warming is an example. Although most experts in the field of climate change state that global warming is created by humans, some scientists do not agree. One of the main problems in these risky situations is – question of responsibility. The world governments should not leave all responsibility to scientists and experts. Authorities should consult experts to declare state of emergency. A strong political initiative is necessary to start dealing with serious ecological problems such as global warming or local environment pollution. Highest level political agreements achieved within the Kyoto Protocol are not enough to stop these phenomena. Clean technologies designed to provide superior performances at lower prices, with lowering losses of conventional offerings – have great chances to be the next driving force to ensure economy growth.

Science is the first to define problems of Earth and life survival. Science is trying to provide solutions, limited by political, social, economic and technology factors. Preservation of life on Earth is the common priority.

Science and technical and technology development can contribute in several fields:

- renewable power sources;*
- efficient energy usage;*
- waste reducing;*
- harmfulness of waste mitigation;*
- recycling;*
- soil, water and air purification;*
- residual waste neutralization.*

Important factor for political decisions making is the public opinion. Therefore, it is extremely important to raise awareness and widely educate population on necessary transition to renewable, ecologically acceptable power sources, which is one of long-term goals of this Conference.

For eight time, this international event is organized by the Society of Renewable Electrical Power Sources (DOIEE) within the Union of Mechanical and Electrotechnical Engineers and Technicians of Serbia (SMETS).

*Belgrade,
October 2020.*

PREDGOVOR

Ubrzani napredak nauke, tehnologije i industrije dovodi do poboljšanja kvaliteta ljudskog života, ali i do stvaranja novih rizičnih situacija. Čovečanstvo je suočeno sa rizicima kakvih u ranijoj ljudskoj istoriji nije bilo. Globalno zagrevanje je tipičan primer. Iako većina eksperata koji proučavaju klimatske promene tvrde da globalno zagrevanje postoji i da je čovek taj koji ga uzrokuje, postoje naučnici koji dovode u sumnju takve tvrdnje. Jedan od glavnih problema vezanih za nove rizične situacije jeste – pitanje odgovornosti. Vlade država u svetu ne smeju teret odgovornosti prepustiti isključivo naučnicima i ekspertima. Vlasti treba da se konsultuju sa ekspertima i da dobro procene kada treba proglasiti opasnost od rizične situacije. Potrebna je jaka politička inicijativa da bi se počeli rešavati ozbiljni ekološki problemi kao što je globalno zagrevanje, ali i lokalno zagađenje životne sredine. Politički dogovori na svetskom nivou koji su do sada postignuti u okviru Kjoto protokola, nedovoljni su za zaustavljanje ovog fenomena. Čiste tehnologije – tehnologije koje su dizajnirane da obezbeđuju superiorne performanse za nižu cenu dok istovremeno kreiraju manji gubitak od konvencionalnih ponuda – imaju velike šanse da budu sledeća motorna snaga koja će obezbediti ekonomski rast.

Nauka, naravno, pre svih uočava probleme opstanka planete i života na njoj. Ona takođe pokušava da ih reši i uspeva onoliko koliko je to realno moguće, imajući u vidu političke, socijalne, ekonomske i tehnološke faktore. Može se konstatovati da su praktično svi prioriteti posvećeni očuvanju života na Zemlji. Nauka i razvoj tehnike i tehnologije mogu tome doprineti u više segmenata:

- obnovljivi izvori energije;*
- energetska efikasnost;*
- smanjenje količine otpada;*
- smanjenje štetnosti otpada;*
- reciklaža;*
- prečišćavanje zemlje, vode i vazduha;*
- neutralizacija preostalog otpada.*

Bitan faktor za donošenje političkih odluka je i javno mnjenje. Zato je jako važno podizanje opšte svesti i što šira edukacija stanovništva o neophodnosti prelaska na obnovljive, ekološki prihvatljive izvore energije, što je jedan od dugoročnih ciljeva ove Konferencije.

Ovaj međunarodni skup po osmi put organizuje Društvo za obnovljive izvore električne energije (DOIEE) Saveza mašinskih i elektrotehničkih inženjera i tehničara Srbije (SMEITS).

*U Beogradu,
oktobra 2020.*

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Zahvaljujući svojim svojstvima, kordierit, $2\text{MgO}\cdot 2\text{Al}_2\text{O}_3\cdot 5\text{SiO}_2$, danas je atraktivan keramički materijal za razne primene, ali sa vrlo visokom temperaturom sinterovanja. Mehanohemijaska aktivacija smeše početnih komponenti izvedena je da bi se snizila temperatura sinterovanja. DTA metoda je korišćena za praćenje temperaturnih promena u analiziranom trokomponentnom sistemu. Kako je ranijim istraživanjima utvrđeno da vreme relaksacije može da utiče na aktivirane komponente u smislu hemijskih promena i stepena aktiviranosti, bilo je značajno utvrditi ima li uticaj i na posmatrani aktivirani system. Uticaj vremena relaksacije na smešu aktiviranih komponentata analiziran je FT IR spektroskopijom i početnih komponenti i aktivirane smeše nakon 24h i 24 meseca perioda relaksacije.

Ključne reči: kordierit; sinterovanje; mekanohemijaska aktivacija; DTA; FT IR

Due to its properties, cordierite, $2\text{MgO}\cdot 2\text{Al}_2\text{O}_3\cdot 5\text{SiO}_2$, is nowadays an attractive ceramic material for various applications but with very high sintering temperature. Mechanochemical activation of the initial components mixture was performed in order to decrease the sintering temperature. DTA method was used to monitor the temperature induced changes in the analyzed three-component system. Since previous research has pointed out that the relaxation time can influence the activated components in terms of chemical changes and the activation degree, it was important to determine whether it has an impact on the observed activated system. The influence of the relaxation time on the activated components mixture was analyzed by FT IR spectroscopy of both the initial components and the activated mixture after 24h and 24 months relaxation periods.

Key words: cordierite; sintering; mechanochemical activation; DTA; FT IR

1 Introduction

Cordierite, one of the most important phases of the $\text{MgO}\text{-SiO}_2\text{-Al}_2\text{O}_3$ system, has a low thermal expansion coefficient, excellent thermal shock resistance, low dielectric constant, high volume resistivity, high chemical durability, and relatively high refractoriness and mechanical strength. This ceramic material is widely used in electronics, for honeycomb-shaped catalyst carriers in automobile exhaust systems, substrate material in integrated circuit boards, and also as a refractory material, owing to its stability at high temperatures [1–3].

Cordierite ceramics can be prepared by conventional sintering methods, but it is difficult to sinter cordierite because of the narrow and sintering range (1300–1400 °C) just below its incongruent melting point [4–6]. The preparation of a homogeneous and fine cordierite powder that can be produced without sintering aids is considered to be highly desirable, due to the limiting factors of additives [7–15]. The thermodynamic principles of the kinetics of the syntheses of cordierite ceramics are given in the literature [5].

Mechanochemically activated samples have more accumulated energy compared to the inactivated initial components. Bearing this in mind, it was important to analyze the possible chemical changes of the activated system after certain periods of time (relaxation period) as they can have an

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influence on the kinetics of the sintering process. The accumulated energy can induce surface and bulk chemisorption of components in the atmosphere. If no changes of an activated sample occurred during the relaxation time, then a mechanochemically activated sample can be sintered after an unlimited period of time.

The aim of this research is to decrease the sintering temperature of cordierite by mechanochemical activation of the initial mixture (caoline (Al_2O_3), quartz (SiO_2), and alkali magnesium carbonate). The free surface of the initial powders increases during the mechanochemical activation and changes in the structure of the material are induced by mechanical energy. The influence of the relaxation time of this mechanochemically activated mixture on the sintering of cordierite ceramics was investigated.

2 Experimental

In this research the next initial raw materials of technical quality were used: MgO (98.60%), Al_2O_3 (99.19%) and SiO_2 (96.10%). The mixture composed of powdered MgO , Al_2O_3 and SiO_2 in the ratio 2:2:5 was mechanochemically activated for 5, 15, 30, 60, 120 and 240 minutes (samples marked with A1-A6, respectively), in a laboratory cylindrical ceramic ball mill (VEB, model 13x10.5). Non-isothermal thermogravimetry (TG) and differential thermal analysis (DTA) were used to monitor the influence of mechanical activation on the samples. For this purpose, a METZSCH DTA instrument was employed in the temperature range 20 to 1500°C, at a heating rate of 10°C/min.

The effect of relaxation, i.e. aging, of the activated cordierite mixture on the sintering processes was monitored by analyzing the mixture activated for 120 min using FT IR spectroscopy. FFT infrared spectra of the samples were recorded on an FFT IR spectrometer BOMEM-HARTMAN & BRAUN MICHELSON MB-100, in the range of wave numbers 4000-300 cm^{-1} at a resolution of 2 cm^{-1} . Since the samples are very sensitive to the presence of moisture, the method of making a suspension with "NuJol" was used to prepare the samples. The purpose of this analysis was to identify possible chemical changes on the activated surface of the components in the mixture as well as structural changes. A non-activated sample (zero sample) was also analyzed by the same chosen methodology [8] and the obtained results were compared.

3 Results and discussion

DTA method was used to investigate the changes in the activated cordierite system during the sintering process at temperatures up to 1600 °C. The samples activated for 5, 60 and 120 minutes were chosen for this examination.

The obtained DTA curves are presented in Figure 4.

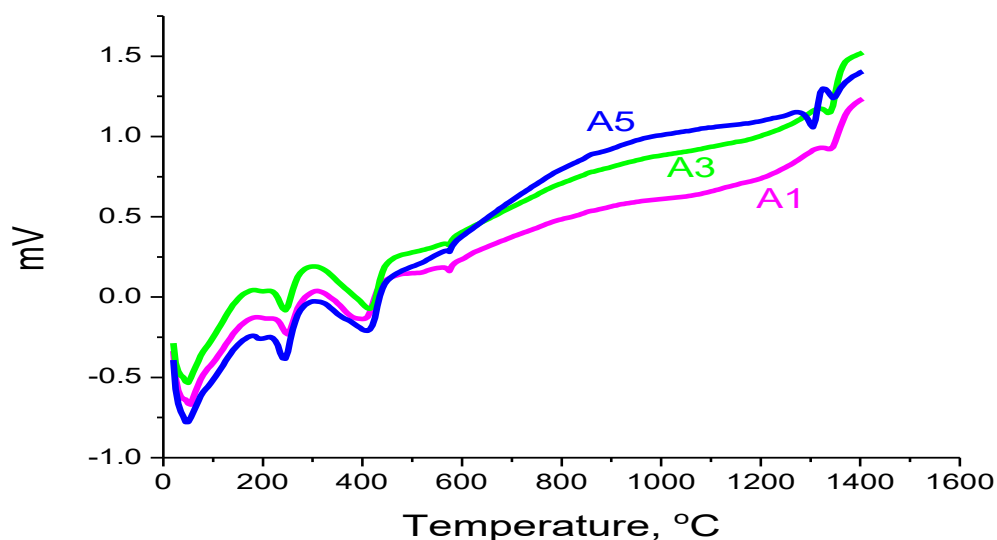


Figure 4. DTA curves of the cordierite mixture activated for 5, 60 and 120 minutes.

As seen in Figure 4, all investigated samples exhibit significant changes up to the temperature of 400 °C, while all the samples behaved quite similarly up to the temperature of 600 °C. Three characteristic peaks are visible: The first peak is up to 100 °C, resulting from loss of humidity. The second in temperature range from 230 to 300 °C which corresponds to the dehydration of $\text{MgCO}_3 \cdot \text{Mg}(\text{OH})_2 \cdot 3\text{H}_2\text{O}$, and the third peak in the range from 390 to 420 °C and it can be attributed to the decomposition of magnesium hydroxyl carbonate.

Indications of the commencement of cordierite formation were detected in the temperature range of 1200-1400 °C for the initial mixture. The corresponding endothermic and exothermic effects were shifted to lower temperatures with increasing duration of mechanical activation.

Derivative of the DTA curve of the sample activated for 5 minutes after a relaxation time of 24 months is presented in Figure 5.

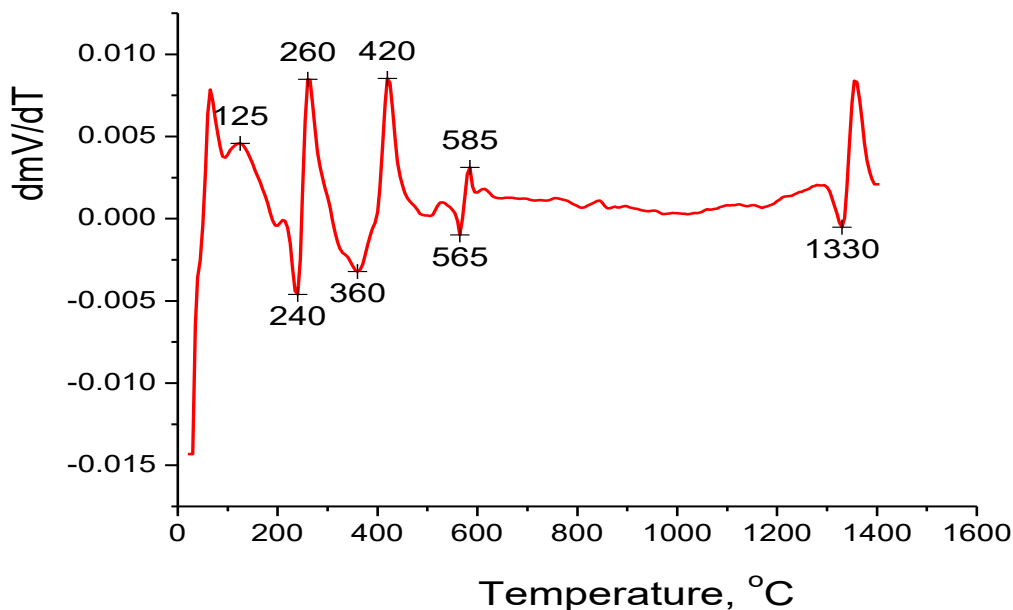


Figure 5. Derivative of the DTA curve for the sample activated for 5 minutes.

By analyzing the curve in Figure 5 it can be noticed that very rapid changes are taking place below the temperature of 600 °C. This indicates transformation reactions in the system at these temperatures. The clearly visible endothermic change at 1315 °C indicates the transformation temperature in the sintering process.

The temperature change of the endothermic effect in the cordierite sintering process from the initial cordierite mixture as a function of the mechanochemical activation duration is presented Figure 6.

As seen in Figure 6, the curve is S-shaped with two plateaus, which are separated by a region of significant changes in the system. The transformation model indicates that the temperature of cordierite sintering decreases with increasing activation time.

The first plateau lasts to 50 minutes of activation, indicating that the changes in the system in this time period were not pronounced enough to have any influence on the sintering process. From 50 to 160 minutes of activation, the changes in the reaction system cause decreasing the sintering temperature. In this range of activation times, it can be seen that the mechanochemical activation had an influence on the kinetics of cordierite sintering, i.e., the activity of the sample increased with increasing activation time. The mechanical energy was used not only for particle attrition but also for increasing the active surface of the particles. This accumulated energy has an influence on the affinity of the components to interact with each other at lower temperatures than in the non-activated systems.

After this period of change, a second plateau appeared when the reactivity of the system ceased to change. Further energy input due to mechanochemical activation results in no significant increase in the reactivity of the system and, hence, there was no further decrease in the cordierite sintering temperature. Thus, prolonging the milling would have no effect.

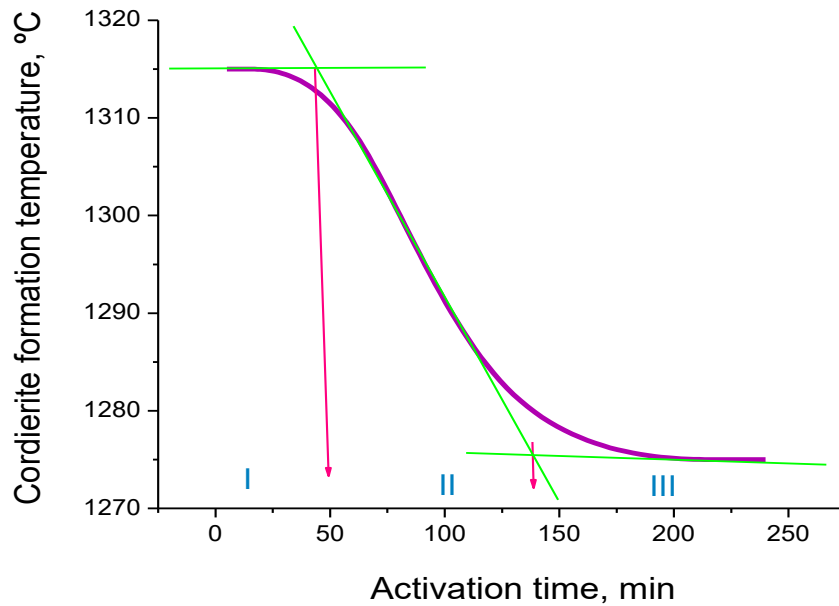


Figure 6. Temperature changes of the endothermic effect in the process of cordierite sintering as a function of the activation time

The temperature decrease of the endothermic and exothermic effects can be described by kinetic equations (Eq. 1):

$$(T_k - T_\infty) / (T_o - T_k) = \exp(-mt) \quad (1)$$

where T_k is the characteristic endothermic or exothermic temperature effect, m is the coefficient of the process, $m = 1.5 \cdot 10^{-2} \text{s}^{-1}$. The endothermic effect favors the transformation of β -quartz to α -quartz, while the exothermic effect results from solid-state reactions between MgO and SiO₂, resulting in the formation of forsterite.

FT IR spectroscopy was used to analyze eventual chemical changes of the activated cordierite mixture during the relaxation time (ambient conditions for up to 24 months). The IR spectra of a sample activated for 120 minutes after relaxation for 24 hours and after 24 months are shown in Figure 7.

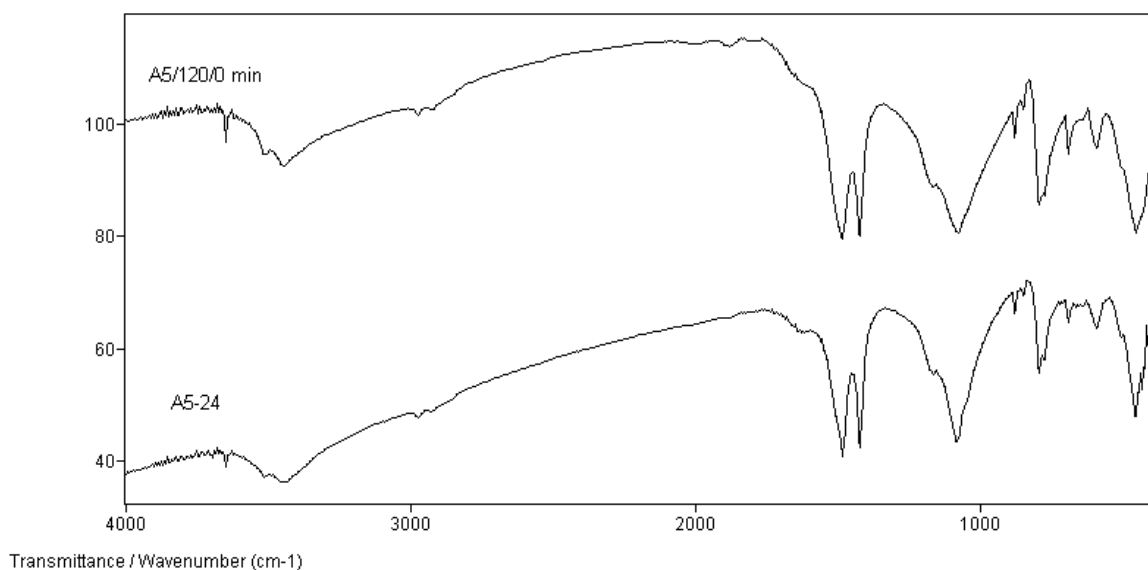


Figure 7. FT IR spectra of sample A5 after relaxation for 24 hours and 24 months.

From the spectra shown in Figure 7, it can be seen that hydroxyl- and carbonate- magnesium compounds were present as impurities in the mixture. The characteristic peaks of bound crystal water

are sharp at the wavenumbers 3445 cm^{-1} , 3512 cm^{-1} and 3649 cm^{-1} . The carbonates are visible at wavenumbers 1425 cm^{-1} and 1485 cm^{-1} , which indicates that the compounds are only impurities due to bound water and carbon dioxide, originating from the atmosphere. Since the initial components are well defined, it is supposed that this analysis proves surface adsorption of humidity and CO_2 from the atmosphere, which resulted in the formation of unstable compounds of hydromagnesite. The IR spectrum of Al_2O_3 shows the existence of $-\text{OH}$ groups at 3443 cm^{-1} , the origin of which originated is air humidity. These results are to be expected since in all experiments technical quality components were used. A relaxation period of 24 months had no influence on the sample since no noticeable changes were visible in the IR spectrum of this sample.

4 Conclusions

DTA proved that the influence of mechanochemical activation of the initial components was caused by an increase in energy of the initial cordierite mixture, which resulted in the endothermic and exothermic sintering reactions being shifted to lower temperatures. The decrease in these temperatures was about 100°C, depending on the activation time.

FT IR analyses showed that relaxation time had no influence on the activated mixtures. It can be concluded that the mechanochemically-activated samples did not change at all up to the moment of sintering, regardless of the activation time. The presented spectra explain the mass losses at the temperatures of 240 and 400 °C, i.e., less-stable compounds (hydroxide and carbonate bonded to hydroxymagnesite) were generated during mechanochemical activation.

Acknowledgments

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