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**9th International Conference on Renewable
Electrical Power Sources**



Beograd, 15. oktobar 2021
Belgrade, October 15, 2021

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obnovljive izvore
električne energije
pri SMEITS-u**

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Za izdavača

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ELEKTRIČNA SVOJSTVA IZOTERMSKI SINTEROVANE KERAMIKE NA BAZI KORDIERITA KAO FUNKCIJA VREMENA SINTEROVANJA I AKTIVACIJE

ELECTRICAL PROPERTIES OF ISOTHERMALLY SINTERED CORDIERITE-BASED CERAMICS AS FUNCTION OF SINTERING AND ACTIVATION TIME

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Mehanička aktivacija je uobičajeno korišćen i relativno brz i jeftin postupak za pripremu uzoraka pre procesa sinterovanja. Kordijerit je stehiometrijska smeša tri različita oksida ($2\text{MgO} \cdot 2\text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2$). To je veoma atraktivan, široko korišćen visokotemperaturni keramički materijal. Mehanička aktivacija početnih smeša sa 5.00 mas. % TiO_2 izvedena je u visokoenergetskom mlinu sa kuglama tokom 10-80 min. Primenjeni pritisak presovanja pre procesa sinterovanja bio je 2 t/cm^2 , na osnovu nedavnog istraživanja. Proces sinterovanja je izvršen na $1350 \text{ }^\circ\text{C}$ tokom 2h i 4h u vazdušnoj atmosferi. Rendgenska difrakcija je korišćena za analizu faznog sastava neaktiviranih uzoraka, uzoraka aktiviranih 80 minuta i sinterovanih 2 i 4 sata, respektivno. Ovaj rad istražuje uticaj produženog vremena sinterovanja na električna svojstva proizvoda.

Ključne reči: mehanička aktivacija; XRD; električna svojstva; kordijerit

Mechanical activation is a commonly used and relatively fast and inexpensive procedure for sample preparation before the sintering process. Cordierite is a stoichiometric mixture of three different oxides ($2\text{MgO} \cdot 2\text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2$). It is a very attractive, widely used high-temperature ceramic material. The mechanical activation of the starting mixtures with 5.00 mass % TiO_2 was performed in a high energy ball mill during 10-80 min. The applied compaction pressure before the sintering process was 2 t/cm^2 , based on our recent investigation. The sintering process was performed at $1350 \text{ }^\circ\text{C}$ for 2h and 4h in air atmosphere. X-ray diffraction was used to analyze the phase composition of non-activated and 80 min activated samples, sintered for 2 and 4h, respectively. This paper investigates the influence of prolonged sintering time on the product electrical properties.

Keywords: mechanical activation; XRD; electrical properties; cordierite

1 Introduction

Mechanical activation is widely used and relatively inexpensive procedure for sintering process sample preparation. Cordierite, $2\text{MgO} \cdot 2\text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2$, is a very actual high-temperature ceramic material, due to its characteristics, and the experiments showed very good answer on mechanical activation. This material is widely used in various fields, from substrates for micro-electronic packaging industry to cookware, heat exchangers, and glazes for floor tiles. Low temperature thermal expansion coefficient ($20 \cdot 10^{-7}/^\circ\text{C}$) and low relative dielectric constant (~ 5), these ceramics makes well known material due to their good thermo-mechanical, chemical and dielectric properties [1,2]. They can be applicable as materials that are exposed to sudden temperature changes [3 -7] and also as a semiconducting bearers [8,9]. The temperature range of cordierite sintering is very narrow ($1300\text{-}1400 \text{ }^\circ\text{C}$).

The experiments showed a significant influence of mechanical activation, as well as compaction pressure on starting mixtures (kaolin, quartz, magnesium oxide) on lowering of sintering temperature [10]. Mechanically activated components increased energy due to induced crystal defects compared

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to non-activated ones. During mechanochemical treatment, several processes occur: attrition of starting material, crystal lattice destruction, various defects formation, etc. All mentioned processes increase the chance that during heating, processes could be observed at lower temperatures than usual [11-18]. Mechanical activation have great influence on final product, and it is important to investigate the possible effect on the final electrical characteristics, so it is very important to approach and understand changes that get introduced into the system during mechanical activation.

In this paper, the authors used starting conditions and study the influence of sintering time on phase composition and electrical properties of sintered samples.

2 Experimental procedure

Mixtures of $Mg(OH)_2$, Al_2O_3 , SiO_2 and TiO_2 (all p.a. purity) were used in these experiments. The cordierite ceramics starting mixture, $MgO+Al_2O_3+SiO_2$ in the 2:2:5 ratio, with the addition of 5.00 mass % TiO_2 , were mechanically activated by grinding in a high-energy planetary ball mill. ZrO_2 vessels and balls were used with the powder to balls mass ratio of 1:40. The milling process was performed in air atmosphere for 10, 20, 40 and 80 minutes. The samples were denoted as K-0, K-10, K-20, K-40 and K-80, according to the milling time.

The pressure used in our experiments was 2 t/cm^2 (approximately 200 MPa). The pressure was performed in a double-sided tool 6 mm in diameter (Hydraulic press RING 14, VEB THURINGER). The compacts were sintered isothermally at $1350 \text{ }^\circ\text{C}$, in air atmosphere for 2h and 4h, with heating rate of $10 \text{ }^\circ\text{C/min}$.

The X-ray powder diffraction patterns after milling and sintering were obtained using Philips PW-1050 diffractometer with $\lambda\text{Cu-K}\alpha$ radiation and a step/time scan mode of $0.05 \text{ }^\circ/1\text{s}$. The morphology of the obtained powders and sintered samples were characterized by scanning electron microscopy (JEOL JSM-6390 LV). The powders were crushed and covered with gold in order to perform these measurements.

3 Results and Discussion

Mechanically activated samples of cordierite with the addition of 5 mass % TiO_2 were sintered for 2 and 4 hours at $1350 \text{ }^\circ\text{C}$. XRD patterns of samples non-activated and activated 80 min sintered at $1350 \text{ }^\circ\text{C}$ for a) 2h and b) 4h is shown in Figures 1 and 2.

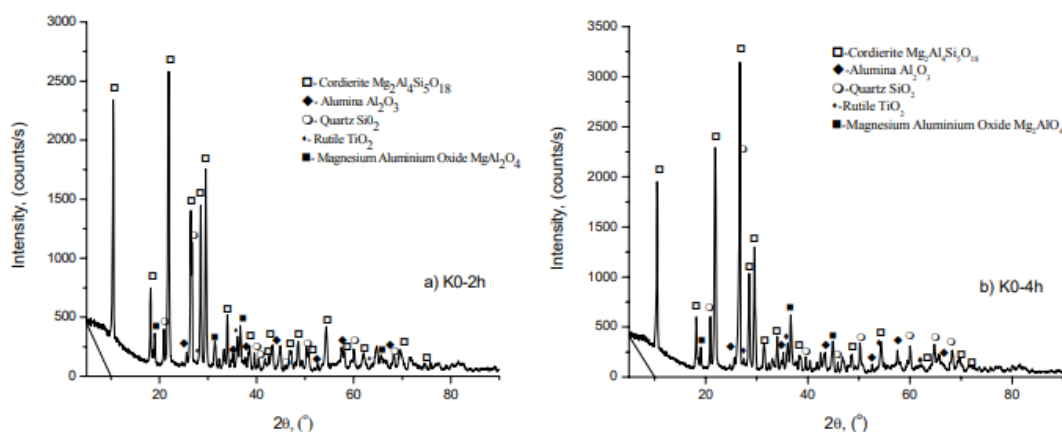


Figure 1. XRD patterns of non-activated samples sintered at $1350 \text{ }^\circ\text{C}$ for a) 2h and b) 4h

XRD analysis of non-activated samples sintered 2 and 4 hours shows a presence of cordierite formation as well as the remains of the starting oxides. Corundum (Al_2O_3), quartz (SiO_2) and $MgAl_2O_4$ and TiO_2 rutile were detected. Comparing the diffraction patterns of non-activated samples, sintered 2 and 4 hours, it can be seen that the sample sintered 4 h have higher amounts of cordierite (66 % compared with 60 % cordierite sintered for 2 hours). The remains of the starting oxides are reduced with the prolonged sintering of the starting material.

Diffraction patterns which are mechanically activated 80 minutes (Figure 2) had significantly greater responses compared to non-activated sintered samples. The sample which was sintered for 2

hours showed practically insignificant traces of the starting oxides corundum (2.4 %) and quartz (0.9 %) and (95.4 %) cordierite phase. The sample sintered for 4 hours has no remaining traces of the starting oxides (except rutile TiO_2 content of 2 % additives whose presence was expected). Cordierite is present in an amount of 98 %, which can be considered a fully completed reaction during the sintering process.

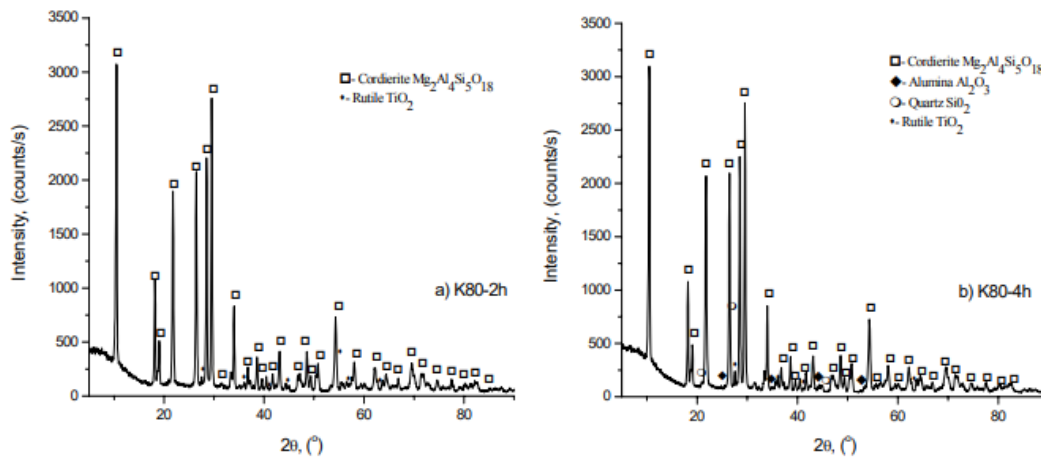


Figure 2. XRD patterns of samples activated 80 min sintered at 1350 °C for a) 2h and b) 4h

Table 1 presents the results of measured capacitance. Electrical properties of the obtained materials were monitored as a function of time of mechanical activation and prolonged sintering time. From these data the value of the dielectric constant was calculated. Electrical measurements were in great accordance with results given previously in the paper regarding XRD patterns. It is obvious that the milling and sintering time have influence on the capacitance and dielectric constant values. Two areas are visible: the first one for samples activated 0 – 20 minutes, and the second for samples activated 20 – 80 min. Both the capacitance and dielectric constant rapidly increase with the first 20 minutes of activation, whereby more significant changes were in the sample sintered for 2 hours. After 20 minutes of mechanical activation changes in capacitance and dielectric constant are smaller. Data value for the dielectric constant of the sintered samples for 2 and 4 hours range from 3.74 to 4.97, as expected.

Table 1. Electrical properties of cordierite after sintering at 1350 °C for a) 2h and b) 4 h as a function of milling time (0 – 80 min)

a)			b)		
2h	C	ϵ_r	4h	C	ϵ_r
0	0.71	3.7418	0	0.77	4.0973
10	0.88	4.6468	10	0.82	4.3248
20	0.88	4.6734	20	0.84	4.5167
40	0.80	4.3363	40	0.92	4.9741
80	0.83	4.6462	80	0.82	4.5992

4 Conclusions

Investigation results shows that sintering time, as well as mechanical activation, has influence on the final characteristics of the sintered material. The results that justify prolonged sintering time showed that at the same sintering temperature, samples activated 80 minutes have a higher percentage of reacted starting material compared to samples that have not been activated. For samples that were sintered 4 hours and 80 minutes activated starting components were not found. Cordierite ceramics

was obtained in an amount of 98%. The remaining 2% is TiO₂ rutile, which was added to the starting mixture as an additive.

Electrical investigation shows the influence of mechanical activation time and sintering time on values of capacitance and dielectric constant. Both capacitance and dielectric constant rapidly increase with first 20 minutes of mechanical activation. Data value for the dielectric constant of the sintered samples 2 and 4 hours range from 3.74 to 4.97, which is in accordance with literature data.

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