

**University of Belgrade  
Technical Faculty in Bor and  
Mining and Metallurgy Institute Bor**



**49<sup>th</sup> International  
October Conference  
on Mining and Metallurgy**

# **PROCEEDINGS**

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**Ljubiša Balanović**

**Bor Lake, Serbia**

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Conference

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49<sup>th</sup> INTERNATIONAL OCTOBER CONFERENCE  
on Mining and Metallurgy**

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## **PREFACE**

On behalf of the Organizing Committee, it is a great honor and pleasure to wish all the participants a warm welcome to the 49<sup>th</sup> International October Conference on Mining and Metallurgy (IOC 2017) held at Bor Lake, Serbia, 18 – 21 October 2017.

The IOC 2017 has been organized by the University of Belgrade, Technical Faculty in Bor, in cooperation with Mining and Metallurgy Institute Bor. It is devoted to presenting recent research results and advances in the fields of geology, mining, metallurgy, materials science, technology, environmental protection, and related engineering topics. The primary goal of IOC is to bring together academics, researchers, and industry engineers to exchange their experiences, expertise and ideas, and also to consider possibilities for collaborative research.

This year's conference is dedicated to the memory of Professor Dragana Zivkovic who was one of our most loyal and active Committee members. The 4<sup>th</sup> International Student Conference on Technical Sciences (ISC 2017) will take place within the frame of IOC 2017. ISC provides a unique opportunity for the students from both the country and the region to promote scientific research and discuss future directions of research with the experts and specialists.

These proceedings include 153 papers from authors coming from universities, research institutes and industries in 30 countries: Austria, Bosnia and Herzegovina, Bulgaria, China, Croatia, Czech Republic, France, Germany, Hungary, India, Iran, Italy, Japan, Jordan, Kazakhstan, Libya, Macedonia, México, Montenegro, Norway, Poland, Romania, Russia, Slovakia, Slovenia, South Africa, Spain, Turkey, USA and Serbia.

Financial assistance provided by the Ministry of Education, Science and Technological Development of the Republic of Serbia is gratefully acknowledged. The support of the sponsors and their willingness and ability to cooperate has been of great importance for the success of IOC 2017. The Organizing Committee would like to extend their appreciation and gratitude to all the sponsors and friends of the Conference for their donations and support.

We would like to thank all the authors who have contributed to these proceedings, and also to the members of the scientific and organizing committees, reviewers, speakers, chairpersons and all the Conference participants for their support to IOC 2017. Sincere thanks to all the people who have contributed to the successful organization of IOC 2017.

We look forward to welcoming you to the 50<sup>th</sup> International October Conference on Mining and Metallurgy (IOC 2018), which will be held in October 2018.

On behalf of the 49<sup>th</sup> IOC Organizing Committee,  
Assistant Professor Ivana Marković, PhD

## IN MEMORIAM



**Prof. dr Dragana Živković**  
(13<sup>th</sup> September 1965 – 26<sup>th</sup> November 2016)

Dragana Živković, a full professor and the dean at the Technical Faculty in Bor, University of Belgrade and a full member of the Academy of Engineering Sciences of Serbia, gave an immeasurable contribution to the development of science and education in the fields of thermodynamics, metallurgical engineering and materials science. She left a deep trace, unique in its nature, not only in Serbia, but also in the world.

Dragana Živković was one of the leading scientists in the field of Thermodynamics of multicomponent metallic systems, Advanced metallic materials, Metallurgy of iron and steel, Kinetics of metallurgical processes and Archaeometallurgy. She published over 200 scientific papers in international SCI journals, over 150 papers in national journals and more than 500 conference papers. Her papers have been cited more than 500 times.

She was involved in about 40 projects, about half of them being international, many of which were coordinated by Dragana herself. She was a member of numerous international and national scientific and professional organizations and associations, the editor-in-chief of Journal of Mining and Metallurgy, Section B: Metallurgy, a member of editorial boards of several international and national journals, the secretary of the Committee of thermodynamics and phase diagrams of Serbia, and the chairman and a member of the scientific and organizing committees of numerous national and international scientific conferences.

Through her continual participation at the International October Conference on Mining and Metallurgy, as an author, as a member of the organizing committee and the president of the scientific committee on several occasions, she managed to make this conference distinguishable in wider scientific circles, connecting people through successful collaboration and lasting friendships.

She was our dear friend, a valued and generous colleague and an inspiring teacher. She touched all of us with her positive attitude, dedication, generosity and friendship.

For all of us who had the privilege to know her, she will always be the part of our lives.

The 49<sup>th</sup> IOC Organizing Committee

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## EFFECTS OF DIFFERENT SINTERING ROUTES ON THE PROPERTIES OF STEATITE-BASED CERAMICS

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### Abstract

*This study provided insights into relations between sintering mechanism and behavior of two types of steatite-based ceramics. Experimental steatite samples were synthesized from low-cost natural raw materials. The sintering was conducted via dilatometer and SPS method. The thermally induced changes in the steatites and its properties were monitored by instrumental analyses: mineral phase transition was investigated via XRD, and the thermal stability was observed using DTA. The investigation highlighted the optimal combinations of the raw materials for production of the steatite ceramics for advanced electrical engineering applications.*

**Keywords:** construction materials; DTA; dilatometry; SPS sintering; insulators.

### 1. INTRODUCTION

The steatite ceramics is, besides cordierite, the main represent of dielectric materials with characteristics such as high mechanical strength, good electrical properties and low dielectric loss [1, 2]. Steatite is a cost-effective alumina replacement, because it satisfies the performance requirements, yet its production is more economical due the facilitated forming [1]. Such predispositions are classifying this material for applications in field of electrical and electronic engineering [3–5]. The steatite ceramic is also employed in medicine as a material for dental implants and artificial bones due to high hardness and good bio-compatibility [6]. Mineral enstatite is natural equivalent of the steatite ( $Mg_3(Si_4O_{10})(OH)_2$ ) [7]. Enstatite has orthorhombic crystal lattice with silica-tetrahedra chains connected by two symmetrical octahedral  $Mg^{2+}$  sites [8, 9]. The steatite ceramics can be manufactured completely from economic natural raw materials, as its mineral base for the synthesis generally comprises the talc mineral mixture for the calcination procedure (80–90 %), plastic clay as a bonding agent (5–10 %) and a melting agent (5–10 %). This mixture of raw materials is fired at temperatures up to 1400 °C depending on the selected sintering procedure [9, 10]. The final structure of steatite is formed though crystallization of mineral phases, and their fusion during the vitrification [10].

This study provides insight into dielectric behavior of steatite that is prepared from natural raw materials (talc,  $BaCO_3$ , feldspar, kaolin) and sintered via dilatometer and Spark Plasma Sintering method. The aim of the study is to detect the critical points in the design of steatite, to make improvements in its synthesis in order to satisfy the increasing demands of the dielectric material performances.

## 2. EXPERIMENTAL

Two steatite mixtures (ST-A and ST-B), were prepared for the experiment. Both steatites were based on talc (80 wt. %). Kaolin clay (10 wt. %) was applied as a bonding agent. The fluxing agents (10 wt. %) were: feldspar in ST-A, and BaCO<sub>3</sub> in ST-B.

Initial sintering curves were constructed from the dilatometric analyses of green bodies recorded at 10 °C/min. The SPS process was carried out in a spark plasma sintering apparatus (Dr. Sinter 2050) in vacuum. Each batch of 2.0 g powder was poured into the cylindrical graphite die with an inner and outer diameter of 12 mm. A uniaxial pressure was automatically loaded to the targeted value of 50 MPa during the initial 3 min and kept constant to the end of each sintering cycle. The samples were firstly automatically heated to 600 °C for 3 min and then to 1000 °C at a heating rate of 100 °C/min. Mineralogical analyses were performed on the pulverized sintered steatite samples by X-ray powder diffraction technique (Philips PW-1710). Differential thermal (DTA) analysis was carried out in a static air flow with an automatic thermal analyzing system Setsys, SETARAM Instrumentation, Caluire, France. The steatite green powdery mixtures (approx. 30 mg) were loosely packed into an alumina holder and thermally treated at a constant heating rate of 10 °C/min in the temperature range between 25 °C and 1000 °C.

## 3. RESULTS AND DISCUSSION

The first stage in the experiment was to monitor the sintering induced changes within mineral phase compositions of the steatite mixes with respect to the employment of different fluxing agents. The thermal treatment of the ST-A steatite conducted at 1000 °C resulted in following mineralogical composition: enstatite, quartz, plagioclase, pyrope, and forsterite. Enstatite and quartz were the most abundant phases. Two additional crystalline phases appeared at 1200 °C: cristobalite and tridymite. Unlike the ST-A steatite, the ST-B steatite showed a wider variety of crystalline phases at the initial sintering temperature. At 1000 °C, the following phases were identified: enstatite, pyrope, forsterite, cristobalite, tridymite,  $\alpha$ -cristobalite,  $\alpha$ -BaAl<sub>2</sub>Si<sub>2</sub>O<sub>8</sub>, and sanbornite. The mineralogical phases such as  $\alpha$ -BaAl<sub>2</sub>Si<sub>2</sub>O<sub>8</sub>, Ba<sub>2</sub>SiO<sub>4</sub>, BaSiO<sub>3</sub>, and sanbornite were all related to BaCO<sub>3</sub> which has been applied as the flux in the composition of this ceramic material. These phases were not present and/or detectable at 1200 °C. The most abundant phase was enstatite. BaCO<sub>3</sub> enabled quicker and more efficient quartz transition. Analogous to the diffractograms of the sample ST-A, the crystallinity of the present mineral phases in ST-B intensified with increasing of the sintering temperature. The majority of reflections on diffractograms at 1200 °C, in case of both steatites, corresponded to the stable enstatite.

The assessment of the thermal behavior of the green steatite powder mixes with respect to the employment of different bonding agents and flux pertained to the second stage of the experiment. The dilatometric curves of the samples ST-A and ST-B are illustrated in Figure 1. The results of the Spark Plasma Sintering (SPS) method are given in Figure 2, and differential thermal curves are shown in Figure 3.

The dilatometric (Figure 1) and DTA curves (Figure 3) of both steatites comprised three stages. The SPS analysis (Figure 2) observed the thermal behavior of the steatites above 600 °C. The results obtained with these three methods lead to mutual conclusions. Since the investigated steatites were primarily based on talc, the identified effects and thermal behavior are related to the talc dehydration. Talc is characterized by the presence of both adsorption water and hydroxyl groups that constitute the space lattice elements. Water is normally driven off during three stages dehydration (115–200 °C; 350–500 °C, and 600–1050 °C). The thermal analyses of the ST-A and ST-B samples registered the first dehydration stage as an endothermic valley in the interval 20–200 °C, without presence of a noticeable peak. The second stage was not recognized on the

DTA curve, because the original mineral composition of talc and its genuine behavior have been altered by the addition of bonding agent and flux.

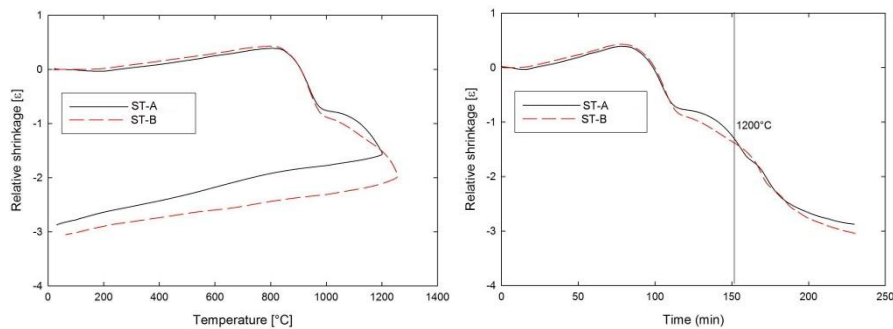


Figure 1 – Dilatometric curves of steatites ST-A and ST-B

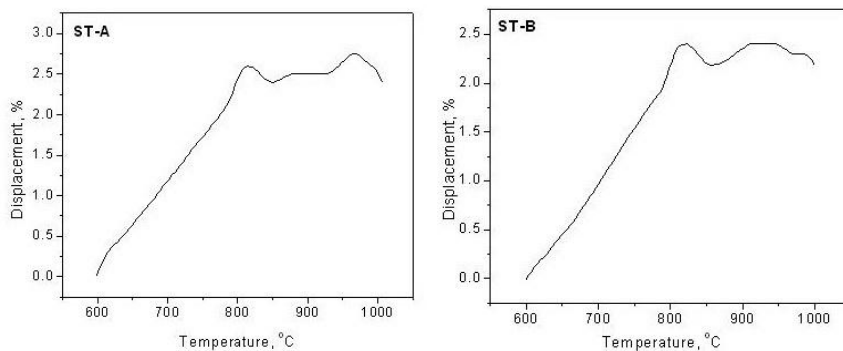


Figure 2 – SPS curves of steatites ST-A and ST-B

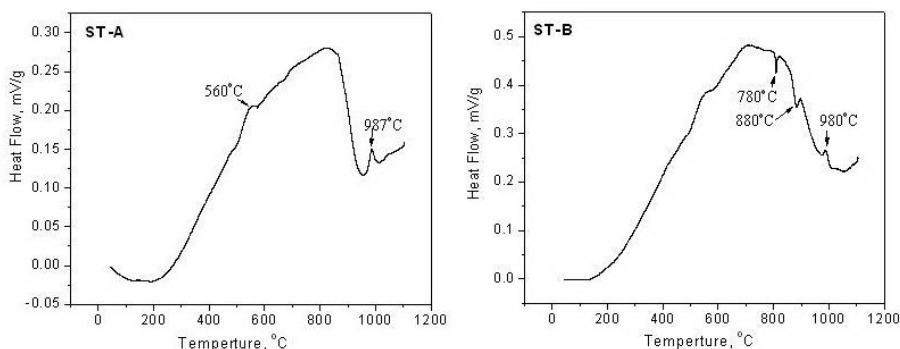


Figure 3 – DTA curves of steatites ST-A and ST-B

During the first two stages only 0.4 % of water was liberated. Therefore, the corresponding peaks were hardly noticeable and/or small. Since 5.1 % of the total non-hygroscopic water was liberated during the third stage, more notable effects were present in this section. The first effects (above 500 °C) were small and hardly detectable. These effects can be related the quartz transformation. Namely, ambient-temperature quartz modification ( $\alpha$ -quartz) reversibly transforms into  $\beta$ -quartz at 573 °C. Since the quantity of quartz was moderate, this resulted in relatively unnoticeable changes in the crystalline structure of steatite. The second effect (sample ST-B), with the peak located at approximately 780 °C, was slightly more notable. The third endothermic effect was recognized as the most prominent one, and it was situated at 987 °C and 980 °C for ST-A and ST-B, respectively. The second and the third peak can be related to the crystallization of SiO<sub>2</sub> as cristobalite, and the crystallization of magnesium metasilicate, respectively. Above 650 °C, talc decomposed into magnesium oxide and silica due to the liberation of constitution water. This reaction would take place at 800 °C in pure talc; however the addition of flux decreased the temperature of decomposition for more than 100 °C. At

1100 °C, the decomposition of pure talc leads to the formation of MgSiO<sub>3</sub>, i.e., protoenstatite (i.e., stable enstatite modification) and amorphous SiO<sub>2</sub>. The temperature of 950 °C can be accepted as the initial stage of stable enstatite formation. Further heating up to 1200 °C contributed to the creation of a well-ordered crystalline structure and establishment of the stable enstatite polymorph as the predominant mineral phase in the steatite composition. The baseline of the ST-B steatite DTA diagram was set significantly higher than ST-A, due to a high portion of amorphous phases formed by addition of a combination of BaCO<sub>3</sub> as flux and kaolin clay as binder.

#### 4. CONCLUSION

The interconnections of sintering procedures and the changes in the behavior of two steatite types were observed. The changes in the densification were induced by the alternations of flux. The XRD analysis highlighted the tendency of steatite mixtures for establishing of the enstatite mono-phase system. The increasing temperature promoted formation of a stable polymorph of enstatite (i.e., protoenstatite) as the main mineral phase. The addition of feldspar did not induce changes in the mineral composition unlike BaCO<sub>3</sub>. The thermal analyses identified a partially modified three-stage dehydration that is characteristic for talc. The recorded effects located above 890 °C were related to the re-crystallization within the talc structure and formation of the stable enstatite polymorph. The shape of DTA curves pointed out to the appearance of an amorphous phase in 500–900 °C range. The glassy matter stabilized newly formed protoenstatite crystals which contributed to the prevention of cracking and deterioration. By observing the crystallinity and thermal behavior it can be concluded that talc – feldspar – kaolin clay is the optimal combination of the raw materials for production of the steatite ceramics with acceptable properties for practical electro-technical applications.

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