

11<sup>TH</sup> CONFERENCE FOR YOUNG SCIENTISTS IN CERAMICS



# **11<sup>TH</sup> C O N F E R E N C E**

## **FOR YOUNG SCIENTISTS IN CERAMICS**

Satellite event:  
**ESR COST IC1208 Workshop**

# **BOOK OF ABSTRACTS**

October 21-24, 2105  
Faculty of Technology  
Novi Sad, Serbia

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## **PROGRAMME and BOOK OF ABSTRACTS**

**October 21-24, 2015  
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coefficient, and low oxygen permeation constant. Thus, yttrium silicate has potential applications as a high-temperature ceramic and environmental/thermal barrier coatings for structural Si-based ceramic materials ( $\text{SiC}$ ,  $\text{Si}_3\text{N}_4$ ).

However, due to the difficulties in the preparation and sintering of yttrium silicate polycrystals, their bulk properties have not yet been investigated. For that reason, fundamental data regarding high temperature mechanical behavior such as creep, strength, toughness or fatigue resistance are not yet available. This data is invaluable to assess the long-term behavior and lifetime of yttrium silicate based on environmental barrier coatings.

The objective of this work is to develop a feasible procedure to produce dense and single phase of  $\text{Y}_2\text{SiO}_5$  polycrystals in order to evaluate these aforementioned properties. To this end, firstly we have synthesized  $\text{Y}_2\text{SiO}_5$  by solid-liquid state reaction using appropriate amounts of  $\text{SiO}_2$  and  $\text{Y}_2\text{O}_3$  powders as precursors and studying the influence of different additives to enhance the synthesis and sintering process by means of the formation of transient liquid phase. Once achieved this aim, we will proceed to evaluate the thermo- mechanical properties.

The phase identification, morphology and microstructure observations of the resulting reaction products were performed using X-ray diffraction (XRD) and scanning electron microscopy (SEM), respectively.

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#### **SYNTHESIS AND CHARACTERIZATION OF RESORBABLE CALCIUM PHOSPHATE BIOCERAMICS WITH A RATIO OF $0.5 \leq \text{Ca/P} \leq 1$**

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Modern regenerative medicine requires resorbable bioactive materials for bone implants. Resorption ability of materials based on calcium phosphates observes at  $\text{Ca/P} < 1.67$  and increases with a decrease of this ratio.

The aim of our research activity was to obtain and explore powders and ceramics based on calcium phosphates with a ratio of  $0.5 \leq \text{Ca/P} \leq 1$ . Five powders were investigated in this range with a ratio of  $\text{Ca/P} = 0.5$ ; 0.625; 0.75; 0.875 and 1.

Solutions of poly- or/and pyrophosphoric acids were obtained from sodium poly- or/and pyrophosphates by means of the ion exchange. Then, calcium nitrate solution was added to these solutions of the acids. Afterward, ammonia solution was added to solutions containing the following ions: pyrophosphate ion, polyphosphate ion,  $\text{NO}_3^-$  and  $\text{Ca}^{2+}$ . In all cases pH-value of these solutions were adjusted to 10. This procedure resulted in formation of amorphous precipitates of hydrated calcium phosphates. Finally, dried and disaggregated powders were pressed into pellets, which were fired to obtain ceramics.

According to thermogravimetric analysis the total weight loss on heating from 20 to 1000 °C for all samples was in the range from 16 to 24%. The weight loss occurred in

two stages: the first stage which took place from 40 °C up to 200 °C associated with the removing of adsorbed water and the second stage (from 200 to 600 °C), associated with the removing of crystallization water and decomposition of ammonium nitrate. Molecular formulas of synthesized hydrated phosphates were deduced from these data as  $\text{Ca}(\text{PO}_3)_2 \cdot \text{H}_2\text{O}$  and  $\text{Ca}_2\text{P}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ .

According to XRD analysis of samples of ceramics, it has been found that for all examples, except the 100% content of pyrophosphate, the ratio between polyphosphate and pyrophosphate pointed before ion exchange was not saved after calcination. Amount of calcium pyrophosphate phase grew in comparison with expected values with increasing Ca/P in the samples. This phenomenon was probably associated with possible thermal hydrolysis of calcium polyphosphate during heat-treatment.

The density of the samples decreases, and the porosity increases with increasing of the content of calcium polyphosphate phase. Despite the low density, these samples demonstrated sufficient strength due to the presence of, calcium polyphosphate forming the melt during sintering. According to SEM micrographs the samples with the high content of polyphosphate had the network of interconnected pores.

The powders of calcium phosphates with a ratio of  $0.5 \leq \text{Ca/P} \leq 1$ , which were obtained by means of the wet precipitation method from solutions of condensed phosphate acids prepared by ion exchange method, had rather uniform composition. Such ceramics consisted of calcium poly- and pyrophosphates are suitable for making bone implants due to high resorption and osteoconductivity.

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#### **SIMULTANEOUS THERMAL ANALYSIS AND DILATOMETRIC STUDY OF HAp-LiFePO<sub>4</sub> SYSTEM**

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Sintered hydroxyapatite bioceramics have been widely studied as a potential material for bone tissue reparation, however, concerning its microstructural and mechanical properties some limits were achieved at the moment. Addition of other materials that could improve functionalities, while preserving inherent advantages of this bioactive ceramics is desirable strategy. In this work, a new idea of addition of lithium iron phosphate as hydroxyapatite sintering aid, provoking liquid phase sintering in the intermediate sintering phase, has been evaluated from the point of view of thermal and dilatometric studies in inert atmosphere, with coupled mass spectroscopy monitoring. Detailed characterization of prepared materials and sintered products is given, confirming the proof of concept. Sintering ability was significantly enhanced and important microstructural features were obtained.