APPLICATIONS OF PHOSPHATE MATERIALS IN THE VESSEL MANUFACTURING MICRO PROPELLANT

Nicolis Amaral de Araújo
Júlia Feldhaus
Instituto Militar de Engenharia - IME
feldhausjulia@gmail.com

Júlio Cesar Soares de Oliveira
Instituto Militar de Engenharia - IME
julio@ime.eb.br

Aldelio Bueno Caldeira
Instituto Militar de Engenharia - IME
aldelio@ime.eb.br

José Carlos Costa da Silva Pinto
Universidade Federal do Rio de Janeiro - UFRJ
pinto@peq.coppe.ufrj.br

Abstract. *The study of prepellants, many materials are used in yhe manufacture of vessel, however, there is a need to seek alternative materials that meet the requirements of a propellant, as well as a good relationship cost benifit. Thus, this study aims at manufacturing a propellant vessel using ceramic coating type phosphate, to create na artificial thermal barrier caused by the combustion generated inside due to the propellant burning, following sintering method with diferente temperatures ranges, and characterized by sacanning electron microscopy (SEM) adding carbono suffering treatment at sputter, and the expansions observed were 1800 and 4000 times. The manufacturing of micro propellant vessel showed satisfactory results with both materials under test, Bellavest SH and Heat Shock brands, after following manufacturing steps: a model is first made of brass, subsequently manufacturing a silicone model using the mold in brass. Then, wax is deposited in the mold to obtain a new model in wax. So is the manufacture of the nozzle made of ceramic material with inserted wax model, and finally the wax casting, obtaining in this way the nozzle with the combustion chamber.*

Keywords: *ceramic material, microcopy, manufacturing.*

1. INTRODUCTION

The materials used as thermal barrier coatings (TBCs) must have the following characteristics: high melting point without phase transformation between room temperature and operation, low thermal conductivity, chemically inert, thermal expansion close to the substrate, good adherence to the substrate and low sintering rate of the porous microstructure (CAO et al., 2004). There are few materials that meet those conditions. Some ceramic coatings such as Al2O3, TiO2, mullite, CaO/MgO + ZrO2, Stabilized zirconia yttria (YSZ), CeO2+YSZ, zircon and La2Zr2O7, etc., they were classified as materials for TBC (CAO et al., 2004).

The ceramic coating is a thermal barrier that reduces the heat flow from the turbulent combustion gases to the substrate. For this reason, the TBC can be transferred to applications in rockets providing thermal shield against oxidation, corrosion and erosion resistance (SCHLOESSER et al., 2010). Furthermore, there is the simplicity of the technology used in the application of TBC.

TBC systems must have a combination of properties to be effective. These ones include low thermal conductivity, high resistance to fragmentation, good erosion resistance, phase stability and morphological stability of pores. In agreement to the study of (MARICOCCHI et al., 1995) for aircraft turbine applications to fragmentation resistance and the thermal conductivity of the coating system is most critical for performance.

Various manufacturing techniques have been developed for micro and meso propellant, for applications at microsatellite propulsion and attitude control. In particular, the manufacturing and technologies of micro thrusters, micro burners are challenging, given the scale and the required accuracy. In addition, new microfabrication technology using ceramic materials with high melting points are relevant to improve thermal stability of the combustor at elevated temperatures (JU and Maruta, 2011).

Currently the technologies adopted to manufacture the structure of a micro propellant are the same used in the manufacture of microelectronic circuit boards. Some of these technologies are based on chemical vapor deposition at low pressure, printing by reactive ion and by deep reactive ion.

The micro thrusters with solid propellant has some advantages over liquid propellant. The micro rocket using solid propellant is simple, with no moving parts and no leakage problems (ORIEUX, 2002; ROSSI, 2002).

The objective of this work was the microestructural analysis by using scanning electron microscopy of the samples of ceramic materials , Bellavest SH and Heat Shock, sintered with different times and temperatures due to evaluate their characteristics to manufacturing a propusor vessel.

2. METODOLOGY

The manufacture tests of the vessel were performed in the Foundry Laboratory and Thermomechanical Treatments - SE / 4 - Military Engineering Institute.

For evaluation of the vessels to be used as a component in micro thruster prototype, tests were made with ceramic material gravitate free and bound phosphate trademark of Bellavest SH, with fast or conventional heating and easy handling with special liquid BegoSol® HE mixture. And with the material for high-melting heat shock, rapid or conventional heating, also with easy handling and control the expansion through the special liquid. The special liquid is a coating liquid bonded with phosphate, being a colloidal silica solution that helps thermal expansion but also increases the resistance of the material before go to the oven.

They are sintered at different temperatures and two time slots, 20 and 30 minutes, according to the minimum and maximum time limits specified by the manufacturer.

2.1 Manufacturing process

The vessels were manufactured from the template shown in Fig 1, the machined material is brass (70 Cu - 30 Zn). Following the technical drawing with the actual dimensions of the model is made a machining operation in a Computational Numeric control machine (CNC) for obtaining the model to manufacturing the propellant vessel, a shaft has being placed on the base to improve handling.

The manufacture of the vessel consists of the following steps:

- Template manufacturing in Elastomer

The elastomer was prepared in powder form, the brand material was Jeltrade® Printer Type II to form a homogeneous paste using about 20% by weight of water which is inserted into a plastic tube covering the entire length of the template body. Then the template is inserted micro thruster metal in the center of the silicone grease tangent to the pipe, awaiting solidification of the silicone paste which lasts about 5 minutes (Fig. 2). Thereafter, the silicon is removed from the tube which surrounds and is carefully cut laterally without reaching the brass template and then it is removed. Therefore, the resultant cavity in silicon is about the micro thruster profile, as can be seen in Figure 3.

The template actual measurements are shown in Table.

Table 1: Measures of micro thruster template in millimeters (mm).

A	B	C	D	D1	D2	D3
11	7,7	2,5	2,6	1,2	0,6	4,4

- Template manufacturing in wax

To obtain the wax template, the wax is melted using a container with rod in “pot-shaped” for handling, the wax is melted by about 100°C on a hot plate (Figure 3). The wax used is of the wax sculpture Newwax® brand. While there is the wax heating, the silicone template is placed on an aluminum plate, this also is on the heating platform, with a piece from a thin wire of the same length of the template, in its inside. This wire helps the neck of template wax does not break, since it is extremely thin; and the goal of simultaneously heating of the template on the aluminum plate is so that the wax does not solidify as runs until the end, which is very common, and let the wax template without the part of the throat.

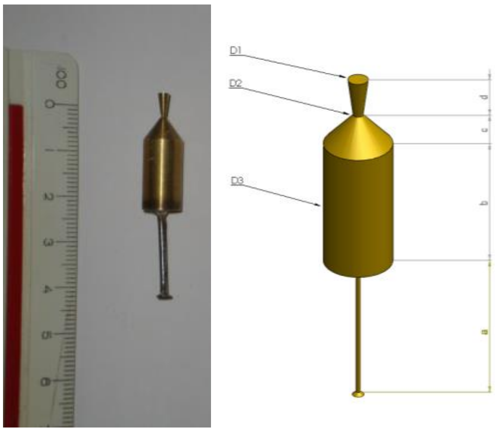


Figure 1: Brass template to the vessel propeller (on the left).

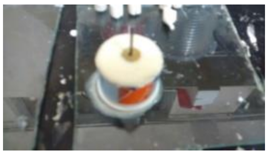


Figure 2: Template manufacturing in Elastomer.

The wax is placed in the cavity formed of silicon thus the solidification was awaited. Then, the elastic silicon wrapped was removed and remains the wax microthruster, as shown in Fig. 4. There is a difficulty in removing the wax inside the silicon, due to the small region of the nozzle throat, which may break during the procedure.

The wax has been used for the templates because they are soft and fragile, and the key property is the thermoplasticity, i.e., they have the ability to soften on heating action. The melting range of them is from 48°C to 90°C.

Figure 3: Elastomer template and wax being melted.

Figure 4: Wax Microthruster template.

- Vessel manufacturing with ceramic material

In this step, the slurry is made with ceramic materials and is inserted around the wax micro propellant, keeping the external arrangement and the free exit of the nozzle. Therefore, awaiting the solidification of the ceramic material for a period of 24 hours in a desiccator to avoid moisture. It is placed inside a furnace at temperature ranging from 100 to 1200°C to evaluate the influence of sintering temperatures for a period of time of 20 and 30 minutes, obtaining a material with the shape shown in Fig. 5.

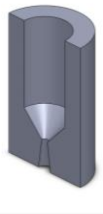


Figure 5: Simplified schematic of a motor vessel (inside view).

2.2 Sintering

The green bodies, with nozzles form, were sintered using sintering temperatures for each of the two samples. The nozzle groups with two materials were heated to temperature: 100°C, 500°C, 900°C and 1200ºC. The four different sintering temperatures were used with the aim to investigate the influence of sintering temperature on the composition of the nozzle with ceramic material.

2.3 Characterization of Materials by Scanning Electron Microscopy (SEM)

The morphology of the material was characterized by a scanning electron microscope (SEM) at the Electron Microscopy Laboratory of IME, Model Quanta 250 both manufactured by Fei Company and operating with maximum voltage of 30kV, high vacuum and secondary electrons. The photomicrographs were processed on an image analyzer from the same manufacturer. The samples before being analyzed in the microscope, suffered addition of a carbon treatment in a metallizer manufactured by Joel, JFC Model 1500. The sizes of "spot" working distance (WD) and expansions of the regions can be observed in the legend of micrographs that will be presented in the chapter on the results.

3. RESULTS AND DISCUSSION

Based on thermogravimetric analysis (TGA), shown in Fig. 6, it can be seen that even the heating track at a maximum temperature of 700°C only two significant losses occur with minimal weight losses in the bands of 100°C may be characterized as water loss and the 200°C range may be volatile compounds of the material composition. From these temperatures the material remains without significant loss of mass may verify their use in the manufacture of sintered propellant vessel in the study of temperatures.

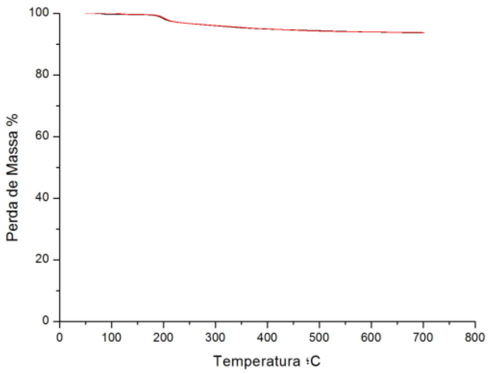
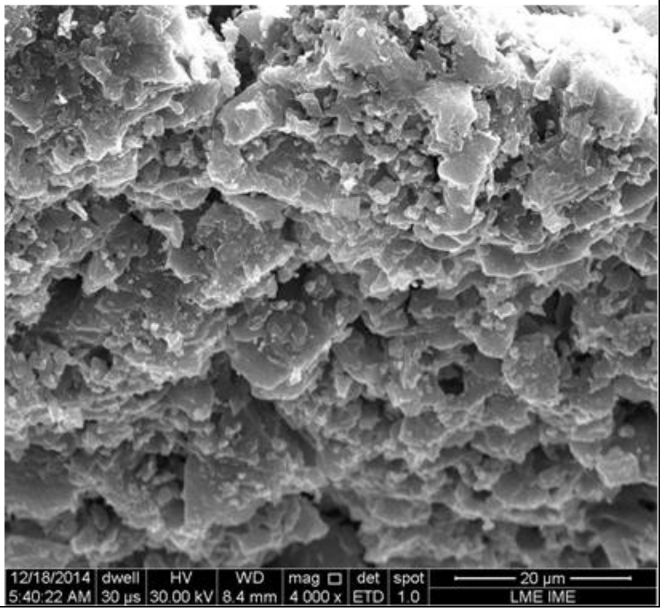
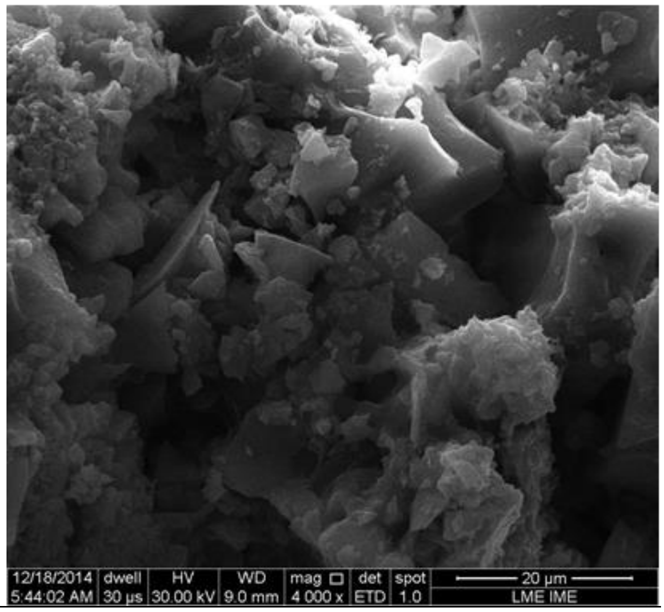


Figure 6: TGA from the used materials (Black– Heat Schock and Red – Bella Vest).

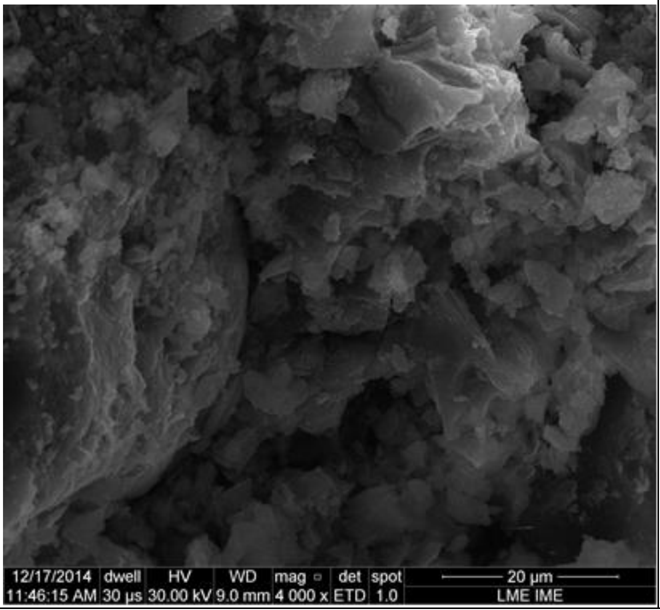
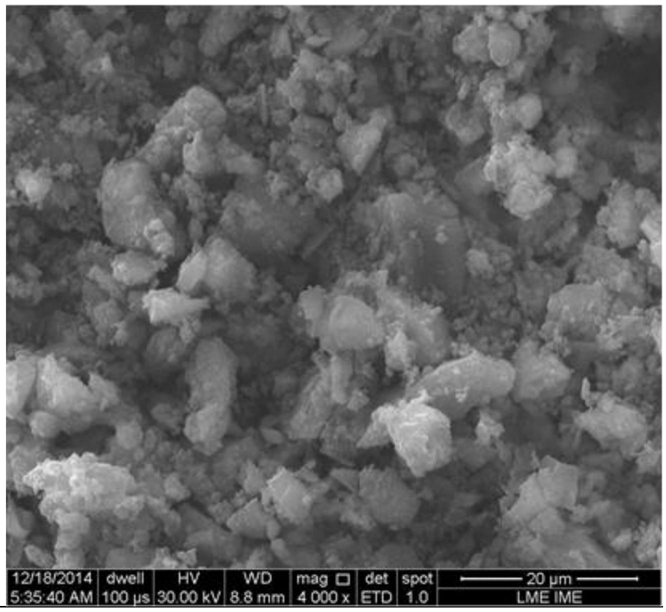
It can be seen that the Bella Vest sample sintered at 100°C shows a rough surface with a high pore fraction and low bond between the particles showing that this temperature was enough only to the initial stages of sintering. The Tab. 2 shows micrographs of sintered samples at 20 and 30 minutes. The sintered sample at 20 minutes has high porosity and small grains, while the sintered sample exhibits at 30 minutes a surface composed of a few pores, smooth and large grains.

Table 2: Microscopy of the samples heated at 100°C.

Tempo: 20 min	**Tempo: 30 minutos**
	

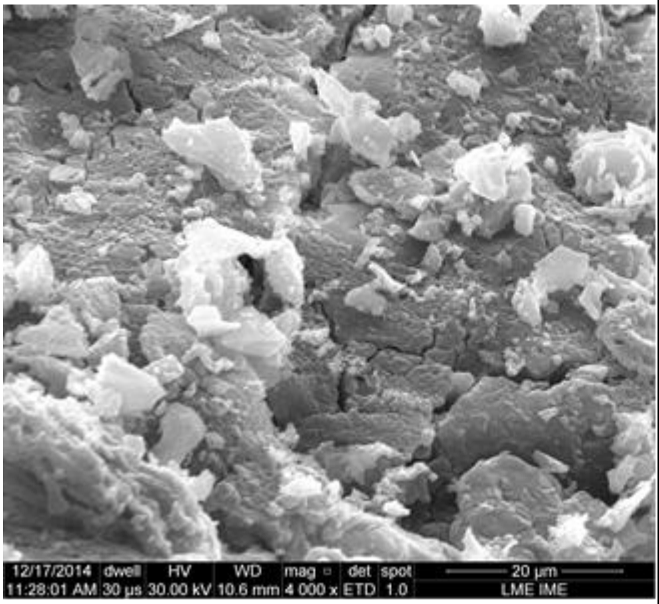
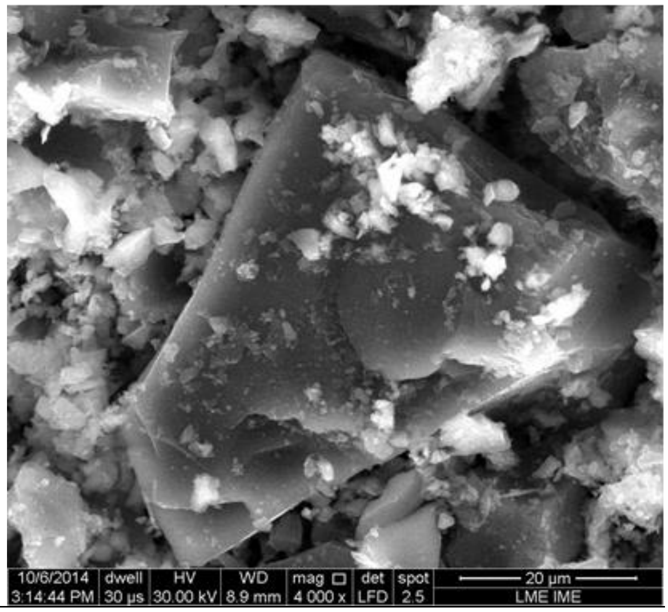
However, comparing the samples in both time it is clear that the period of time has not meaningfully affected in both sintering at 100°C and 500°C, Tab.3.

Table 3: Microscopy of the samples heated at 500 °C.

Tempo: 20 min	**Tempo: 30 minutos**
	

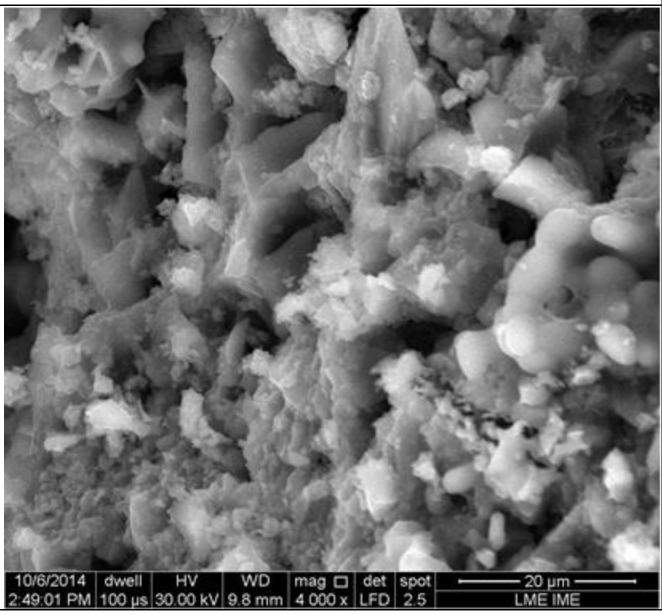
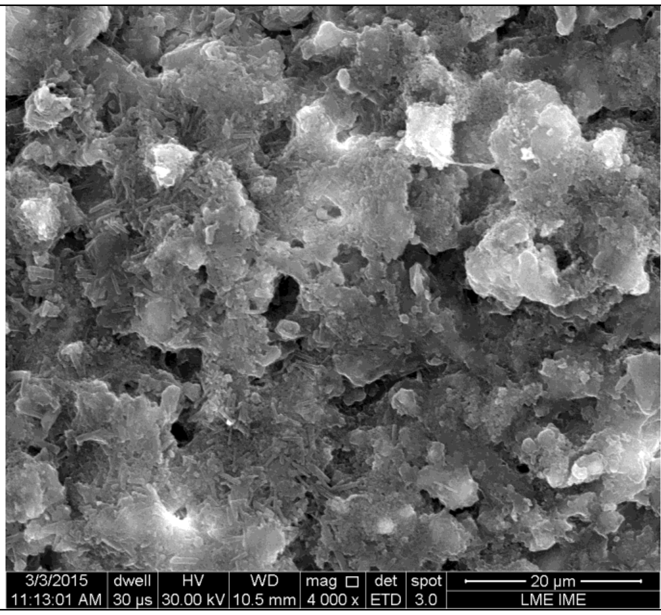
However, at the temperature of 900°C, Tab. 4, it has noticed the emergence of particles of size and appearance significantly higher than previous temperatures of particles under study. This fact may be explained by the difference of sintering temperature, in which provide a higher level of thermal energy thereby increasing the compaction of the material.

Table 4: Microscopy of the samples heated at 900 °C.

Tempo: 20 min	**Tempo: 30 minutos**
	

At 1200°C noticed a microstructure that exposes a significant growth of the generated grains with isolated pores, those characteristics evince that the final stage of sintering at 1200°C was reached (Tab. 5). At this moment, the two materials can be compared at the final sintering temperatures in which noticed that similar behavior occurred during the previous steps with the same temperatures studied.

Table 5: Microscopy of the samples heated at 1200 °C for 30 minutes.

Bella Vest	**Heat Shock**
	

4. CONCLUSION

The results of the scanning electron microscopy showed that the materials has the same behavioral synthesis in the temparatures that was analysed , however in the initital temperatures do not had a efficient synthesis, but, in the temperature of 1200°C occurred high compaction and grain growth. Showing the use of both materials to the manufacturing of prototypes of propusor vessels in test, since that the material shows good fracture resistance and interaction of the materials in the sintering. The times in testo f exhibition to heat do not showed a big influence on results, so it is advisable to the long user in question.

5. REFERENCES

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