# Manufacturing Processes of Paraffin Grains as Fuel for Hybrid Rocket Engines

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# Manufacturing Processes of Paraffin Grains as Fuel for Hybrid Rocket Engines

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**Within the national HYPROB-HYBRID project, funded by the Italian Minister of Education, University and Research (MIUR), the Italian Aerospace Research Centre (CIRA) is carrying out research activities aimed to realize an on-ground demonstrator of Hybrid Rocket Engine (HRE), with the main goal to validate design methodologies and to acquire expertise on enabling technologies and manufacturing processes. This technological demonstrator, with a thrust class of 30 kN, is based on nitrous oxide ($N_2O$) and microcrystalline paraffin wax and will have most attractive capabilities of hybrid systems compared to solid or liquid engines, e.g., throttability and re-ignition.**

**This paper deals with a preliminary methodological assessment and optimization of paraffin grain manufacturing processes. As known, paraffin wax is intrinsically a brittle, thermoplastic material, very sensitive to the presence of surface or internal rips, flaws, voids, micro-cracks and other microstructural high radius defects. Nevertheless, during combustion, it undergoes elevated thermal (temperature of about 3300 K) and mechanical stresses (pressure chamber of 40 bar) which can initiate and propagate deleterious unstable cracks, leading to the catastrophic failure of the grain and consequent danger of combustion instability, up to explosion of the whole rocket. This undesired behaviour strongly compromises one of the most important advantages of HRE in comparison with Liquid Rocket Engines (LRE) and Solid Rocket Engines (SRE), i.e., its inherent safety due to the separate storage and phases of fuel and oxidizer. Therefore it is crucial to select an efficient and reliable manufacturing procedure able to fabricate paraffin wax solid grain free from defects of critical size.**



## Nomenclature

### Acronyms

| | |
|---|---|
| AM | Additive Manufacturing |
| CAD | Computer Aided Design |
| CIRA | Italian Aerospace Research Centre |
| DSC | Differential Scanning Calorimeter |
| FDM | Fused Deposition Modeling |
| FE | Finite Element |
| HRE | Hybrid Rocket Engines |
| HTPB | Hydroxyl Terminated PolyButadiene |
| LRE | Liquid Rocket Engines |
| SRE | Solid Rocket Engines |
| TGA | Thermal Gravimetric Analysis |

### Mathematical symbols

| | |
|---|---|
| $a_a$ | Radius of critical flaw |
| $K_{IC}$ | Stress intensity factor |
| Y | Fracture toughness dimensionless geometric factor |
| $\sigma_c$ | Critical stress |

### Chemical symbols

| | |
|---|---|
| $N_2O$ | Nitrous oxide |

## I. Introduction

Objective of the HYPROB-HYBRID project, pursued by CIRA, is the design and development of an on ground technological HRE demonstrator with a thrust class of 30 kN, operating with gaseous $N_2O$ as oxidizer and solid microcrystalline paraffin wax as fuel.

Among the various fuels for HRE, paraffin wax has the very appealing advantage to possess enhanced burning rates in comparison with other conventional fuels, e.g., Hydroxyl Terminated PolyButadiene (HTPB), due to increased regression rate (3 to 5 time greater than HTPB), mainly ascribed to the formation of a thin, hydro-dynamically unstable liquid layer on the melting surface of the paraffin wax and consequent entrainment of liquid droplets from the gas-solid interface [1], [2] (see Figure 1).

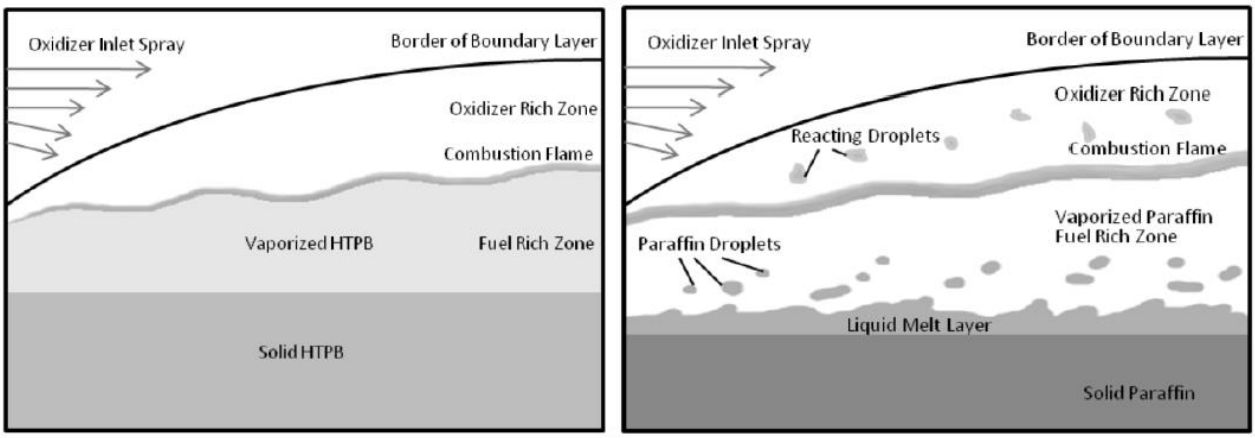


**Figure 1. Schematic comparison between the liquefying and not liquefying solid fuel [3].**

Moreover paraffin wax as HRE solid fuel presents also other interesting advantages over the classical HTPB systems [4], [5]. In details it is non-toxic, non-hazardous, shippable as freight cargo, low cost, reusable (recycling possibilities); it has the same energy per unit mass as kerosene, but its density is 16% greater; it has no scrap possibility, and a long shelf life. Finally, since this fuel is non-explosive, it can be fabricated on-site and thus can save in both manufacturing and launch operation costs [6], [7].

The above mentioned reasons encourage the selection of paraffin wax as optimum fuel for HRE, however this material should be manufactured according to the efficiency and reliability requirements, usually prescribed for the HRE launch and operation, and special care should be paid for avoiding surface and internal rips, defects, micro-cracks and other microstructural discontinuities in order to prevent structural integrity failure due to the following two main reasons:

1. intrinsic brittle nature of this thermoplastic material;
2. volumetric shrinking of about 15 – 25%, depending on the grade of wax, when transitioning from liquid to solid as reported in literature [5].

Moreover for a grain of large size, possible scale effect can appear, mainly due to the increased influence of the thermal gradients inside the grain. This density variation between liquid and solid phases makes fabrication process a real challenge.

### *A. Brief Literature Review*

Usually cylindrical shaped, large volume, hollow paraffin wax solid grains fuels for HRE, with an annular central port, are manufactured by means of laboratory or industrial methods according to the sketch shown in Figure 2.

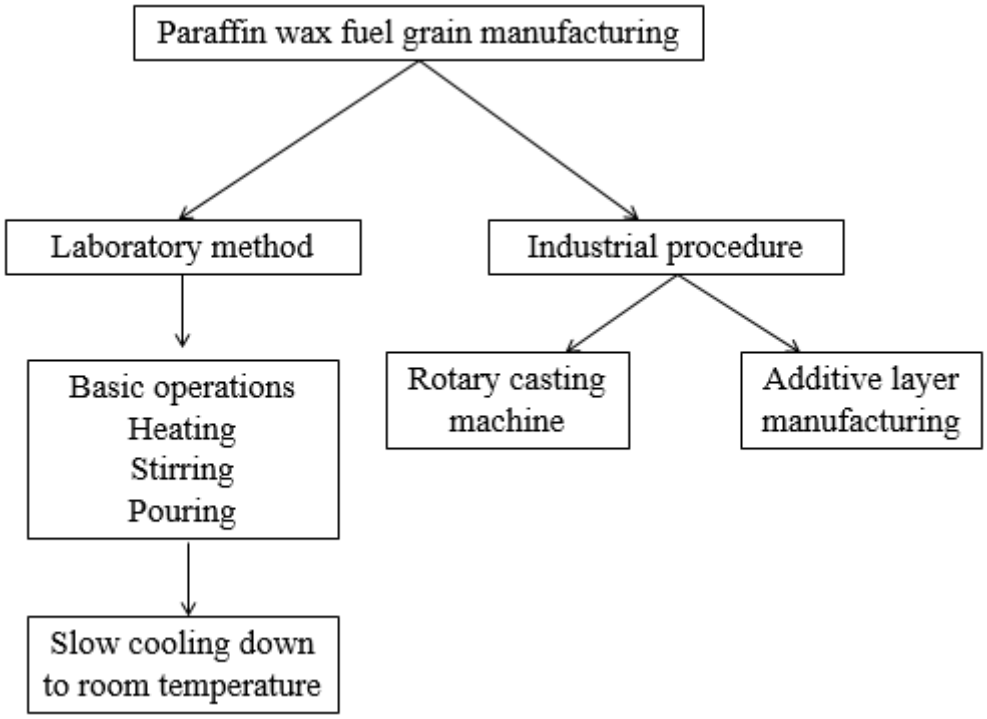


**Figure 2. Sketch of the paraffin wax fuel grain manufacturing processes.**

Some authors have fabricated paraffin wax solid grain fuels for HRE using laboratory based equipment consisting in a circular mould with a piston for applying a mechanical force, able to compensate the volumetric shrinking exhibited by the paraffin wax during transition from liquid to solid state [8].

Other investigators have preferred a more industrial procedure requiring a special spin casting machine with a variable rotational speed, also useful for the creation of the central port [3], [5]. This method implies the operation of an expensive, suitably designed, rotary casting machine, as illustrated in Figure 3, and consists in the following main steps:

- designing a suitable rotary casting machine, which capability must obviously match the size of the hybrid motor fuel grain (in Figure 3 is shown a prototype);
- heating the paraffin wax at a temperature above of the melting point of the system;
- stirring the mixture until a uniform and homogeneous blend is obtained;
- pouring the mixture into the metallic cylindrical combustion chamber;
- capping the ends of the combustion chamber;
- fastening tube in horizontal position;
- spinning the tube around its longitudinal axis at a suitable spinning rate.

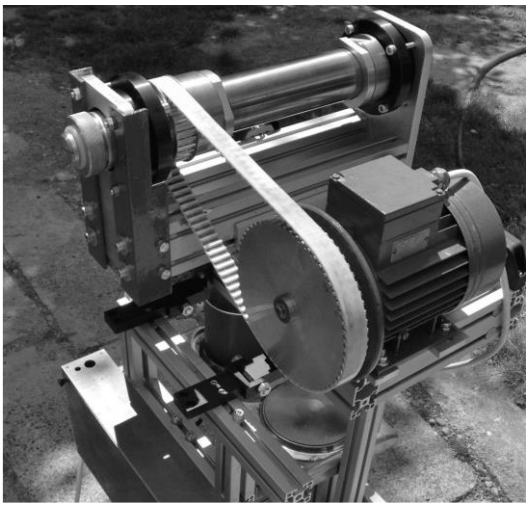

**Figure 3. Rotary wax casting machine [4].**

The central port is formed by the centrifugal force itself during the spinning at an appropriate rate. The solid grain produced according to this method, possesses a glass-smooth surface finish with tolerances of about ±0.7 mm. However, the centrifugal casting process requires an expensive machine and also a not trivial optimization of the manufacturing process parameters, e.g., the spinning rate. Therefore, its use is more justified only in the industry sector for high volume production.

Finally other authors have employed and demonstrated, by means of Finite Element (FE) analysis and experimentation, the effectiveness of a special insulated, aluminum, cylindrical mould, with thermal control for decreasing the temperature gradients inside the paraffin wax grain and therefore reducing the influence of volumetric shrinking and the probability of micro-cracks formation [3]. Here the authors propose the following fuel preparation method:

- heating and melting of paraffin and additives in a tank;
- homogenization with a blending system;
- pouring into a mould made of two aluminium cylinders.

Figure 4 shows a sketch of the manufacturing equipment used.

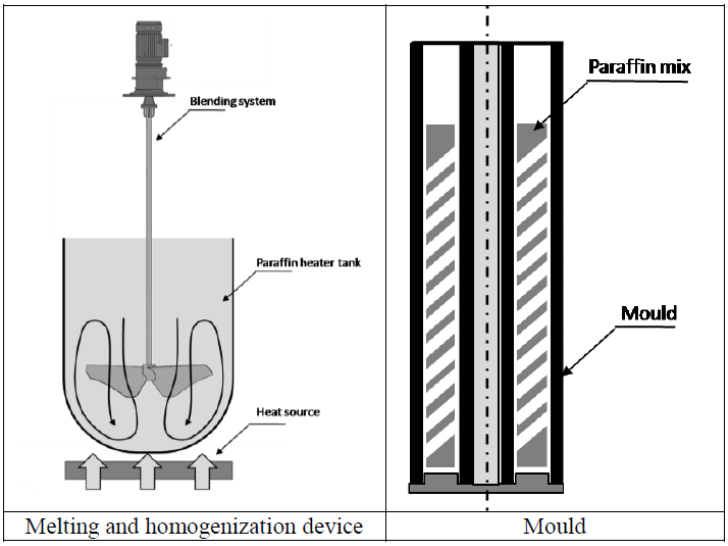


**Figure 4. Casting process selected by Gerbal et al. [3].**

In this case the heat exchange has been allowed only through two external walls, i.e., the top and bottom surfaces, resulting in a very slow cooling rate up to room temperature.

Finally it is under preliminary investigation, in research and industry sectors, the very interesting possibility to combine Additive Manufacturing (AM) methods with HRE design and development for fabricating solid grains of paraffin wax without critical size defects by means of layer-by-layer Fused Deposition Modeling (FDM), 3D printing procedure. This technology offers the advantage of producing homogeneous, solid grains with higher consistency, lower costs and shorter lead-times in comparison with the conventional casting methods with ports of complex geometry and consequent different thrust profiles.

### *B. Rationale*

In the present work the assessment and optimization of paraffin wax solid grain fuels for HRE manufacturing process, as recommended by Gerbal et al. [3], has been performed according to the following fundamental criteria:

- low cost;
- elevated operational easiness;
- small volume production;
- repeatability and reproducibility;
- efficiency and reliability;
- applicable to final demonstrator size grains.

Therefore on basis of the first two criteria, i.e., cheapness and manufacturing easiness, the industrial procedure based on the use of an expensive, suitably designed, rotary casting machine has been discarded and at least at the beginning only laboratory methods requiring basic operations as weighing, heating, stirring, pouring, cooling have been employed.

Since adopting only these simple strategies no satisfactory results have been achieved, special laboratory equipment has been designed and set-up. It consists in the application of both mechanical and thermal energy, i.e., pressure and heat flux, aimed to oppose the effect of volumetric shrinking occurring during cooling and transition from liquid to solid state. This second methodology, which fulfills the above mentioned criteria, has been proved to be very suitable for our purposes. In fact in this way homogeneous, compact, paraffin wax solid grains free from critical size defects have been manufactured.

Finally AM methods was preliminary rejected, because at the moment the building volumes of commercial available 3D printers is limited to an height of about 25-30 cm that is much shorter than the HYPROB-HYBRID rocket engine demonstrator solid fuel grain length i.e., about 80 cm. Therefore AM methods are not immediately applicable to manufacture the final demonstrator paraffin wax solid fuel grain.

## II. Materials and Equipment

### *A. Materials*

The material used in the present investigation is the microcrystalline paraffin wax commercialized by the SASOL® , labelled with the trade code 0907 and predominantly composed of branched alkanes, with a molecular weight centred on the $C_{50}$ value. In Table 1 the physical-chemical properties of SASOL® 0907, as declared by the supplier, are reported, while in Figure 5 the molecular weight distribution is schematically illustrated.

| Wax | Melting or droppling point [°C] | Congealing point [°C] | Oil content [%] | Penetration @ 25°C [1/10mm] | Kinematic viscosity @ 100°C [mm$^2$/s] |
|---|---|---|---|---|---|
| 0907 | 88-102 | 83-94 | 0-1.5 | 4-10 | 14-18 |

**Table 1. SASOL® 0907 paraffin wax properties as declared by the supplier.**

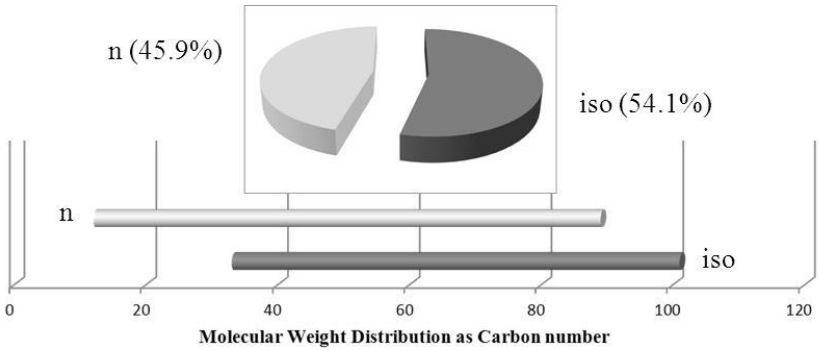


**Figure 5: Molecular weight distribution (higher than the 10 wt% vs the maximum abundance) as carbon number in linear (n) and branched (iso) fractions.**

The high fraction of branched iso-alkanes in the SASOL® 0907 paraffin wax might confer the advantage to form microcrystals, which guarantee more elevated fracture toughness and then a better workability in comparison with the linear paraffin waxes. In fact, according to the Griffith theory (Equation 1) higher fracture toughness $K_{IC}$ allows the application of more elevated critical stress $\sigma_c$ on the solid grain, at constant critical size of internal micro-cracks and of other microstructural defects $a_c$, voids, and rips [9].

$$\text{Eq 1.} \quad \sigma_c = \frac{K_{IC}}{Y(\pi a_c)^{1/2}}$$

where $Y$ is an dimensionless parameter linked to both the configuration of the system and the loading conditions. This is one of the reasons why paraffin waxes are suitable for production of HRE solid grain fuels.

Samples of solid paraffin wax grains of cylindrical shape, with a coaxial central port, have been manufactured according to the geometrical configuration illustrated by Computer Aided Design (CAD) technical draw in Figure 6.

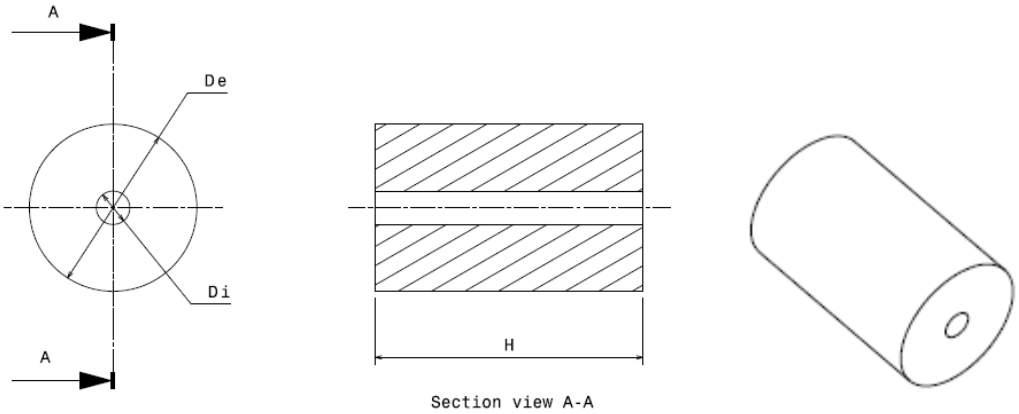


**Figure 6. CAD of paraffin wax solid fuel grain sample configuration.**

Several samples of different sizes, with increasing aspect ratio, have been fabricated. Height, external diameter and internal port diameter of each configuration are listed in Table 2. For the third configuration the central co-axial port has been realized, after solidification, by means of mechanical machining.

| Configuration | Height H [mm] | External diameter De [mm] | Internal diameter Di [mm] | Aspect ratio |
|---|---|---|---|---|
| **1** | 95 | 50 | Not present | 0.5 |
| **2** | 80 | 65 | Not present | 0.8 |
| **3** | 35 | 50 | 4 | 1.4 |

**Table 2. Size of solid paraffin wax grain configurations.**

### *B. Equipment*

The microscope observations have been carried out using a stereo microscope LEICA MZ-12X, whereas the micro-fractography was realized using an optical microscope LEICA DM-RXE in bright field and using an optical magnification of 550x.

Innovative special device, described in more details in section III, has been designed, assembled and used for die casting of paraffin wax solid grains.

## III. Results and Discussions

### A. Preliminary characterization

First of all a preliminary calorimetric and rheological characterizations of the selected paraffin wax have been performed for acquiring useful information for both manufacturing processing assessment and regression rate determination aimed to firing tests [10].

In particular, the Differential Scanning Calorimeter (DSC) carried out at a heating rate of 10°C/min is shown in Figure 7 together with measured parameters reported in Table 3.

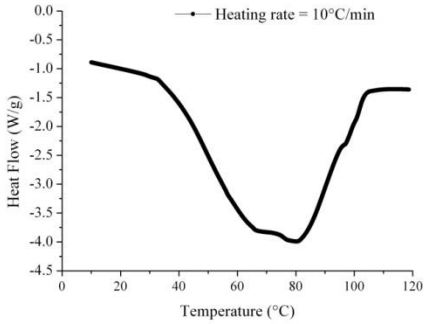


**Figure 7. DSC dynamic scan of SASOL® 0907 at 10°C/min.**

| Heating/Cooling rate [°C/min] | $T_{m,1}$ [°C] | $T_{m,2}$ [°C] | $T_{m,3}$ [°C] | $T_{c,1}$ [°C] | $T_{c,2}$ [°C] | $T_{c,3}$ [°C] |
|---|---|---|---|---|---|---|
| **10** | 67 | 81 | 97 | 54 | 74 | 83 |

**Table 3. Parameters obtained from DSC measurements.**

It highlights that the melting process occurs in the range 30-110°C, with three different melting peaks corresponding to the maxima of the molecular weight distributions of linear and branched alkanes [10]. Results related to the thermo-gravimetric and rheological analyses are listed in Table 4 and in Table 5, respectively, and described in more details in a previous work [10].

| Temperature [°C] | Residual [%] |
|---|---|
| 350 | 23.6 |
| 450 | 9.97 |
| 650 | 0.04 |

**Table 4. Thermal Gravimetric Analysis (TGA) data recorded in air atmosphere at 10°C/min. The temperature of 450°C is assumed as the combustion chamber temperature, corresponding to the evaporation temperature of the paraffin wax 0907.**

| Temperature [°C] | Storage Modulus [MPa] |
|---|---|
| 25 | 1.78 |
| 73.8 | 1.01 |

**Table 5. Rheological properties obtained at a heating rate of 2°C/min and a frequency of 1Hz.**

### B. Preliminary manufacturing assessment

At the beginning, as suggested by literature, the basic laboratory method has been used for the first, preliminary paraffin wax solid fuel grains manufacturing tests. This method consists in the following fundamental operations:

- weighting the required amount of paraffin wax pellets;
- inserting pellets inside a suitable mould;
- heating the pellets to complete liquefying;
- stirring the liquid melt;
- slow cooling the melt down to room temperature.

In the first experiment, weighted paraffin wax pellets have been put inside a polyamide mould, which size and shape corresponds to the configuration 1 in Table 2, then heated up to 110°C to be sure all SASOL® 0907 crystals were melted, as suggested by DSC analysis. The heating process has been carried out using an electric oven under high vacuum, in order to remove gas. Finally the liquid melt has been slowly cooled at about 1°C/min down to room temperature, with the aim to reduce the formation of cracks and voids. No device aimed to apply an external, mechanical pressure on the melt during solidification has been used and very slow cooling was the only expedient employed for decreasing thermal gradients inside the material.

Photographs image of the paraffin wax solid grain sample obtained according to this laboratory method is reported in Figure 8.

Furthermore this sample has been sectioned along the longitudinal axis and the resulting fracture surface has been microscopically analyzed in order to investigate the possible presence of internal micro-cracks, micro-voids and other high radius discontinuities. Results are shown in Figure 9, in Figure 10 and in Figure 11.

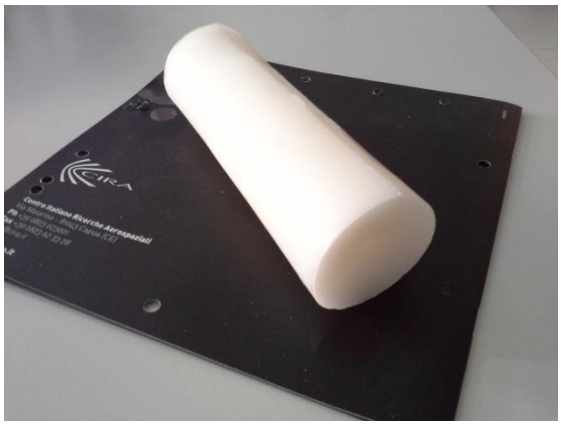

**Figure 8. Paraffin wax solid grain sample manufactured with the laboratory method at 120°C as peak temperature.**

**Figure 9. Fracture surfaces of paraffin wax solid grains manufactured with the laboratory method at 110°C as peak temperature.**

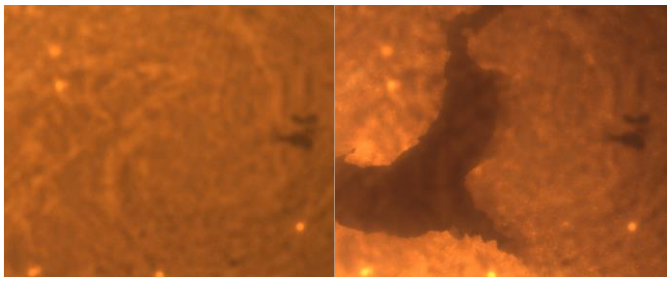

**Figure 10. Optical microscope images of paraffin wax solid grain samples manufactured with the laboratory method at 110°C as peak temperature.**

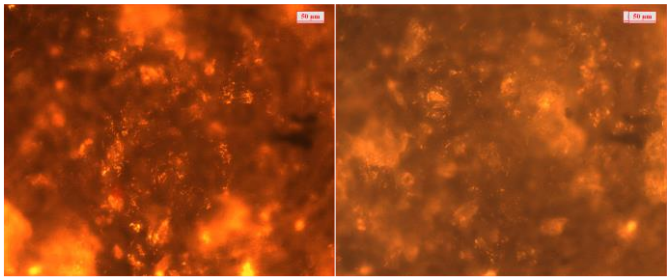

**Figure 11. Optical microscope images obtained in bright field using a magnification of 550x of paraffin wax solid grain samples manufactured with the laboratory method at 110°C as peak temperature.**

Microscopic observations reveal the presence of an internal, large area of macro-porosity, because the spontaneous volumetric shrinkage has not been adequately opposed by an external applied pressure.

Further experiments have been performed, employing a very similar procedure, but changing the material mould, size and shape i.e., aluminum cylindrical vessel associated to the configuration 2 in Table 2. Moreover the peak temperature has been modified in 80°C, which is the temperature corresponding to the maximum absolute value of heat flux during the DSC scan (see Figure 7).

Photograph image of sample manufactured with this second laboratory procedure after sectioning is shown in Figure 12, whereas Figure 13, Figure 14 and Figure 15 illustrate the most important features revealed by microscopic analysis.


**Figure 12. Paraffin wax solid grain sample manufactured with the laboratory method at 80°C as peak temperature.**

**Figure 13. Fracture surfaces of paraffin wax solid grains manufactured with the laboratory method at 80°C as peak temperature.**

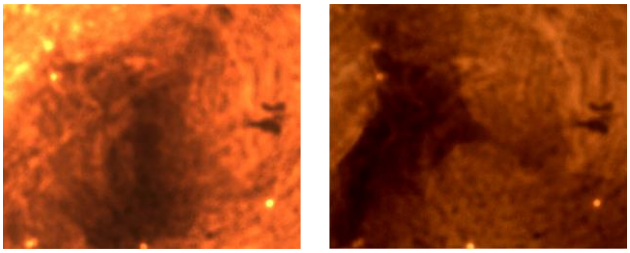

**Figure 14. Optical microscope images of paraffin wax solid grain samples manufactured with the laboratory method at 80°C as peak temperature.**

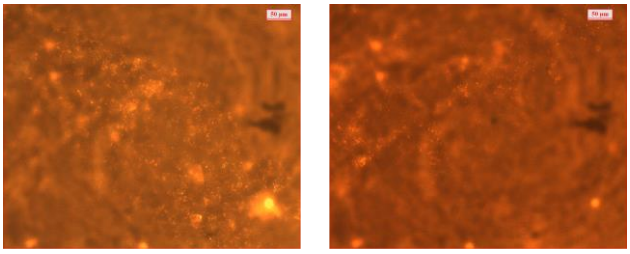

**Figure 15. Optical microscope images obtained in bright field using a magnification of 550x of paraffin wax solid grain samples manufactured with the laboratory method at 80°C as peak temperature.**

From microscopic observations the following considerations can be deduced:

- edges are very homogeneous while the core region shows traces of pellets only partially liquefied;
- internal micro-voids and micro-cracks are present.

The first point is a consequence of improper heating. In fact the peak temperature i.e., 80°C is not sufficient for liquefying the microcrystalline paraffin wax hydrocarbon chains with higher molecular weight. Instead the presence of internal flaws is ascribed to the spontaneous, volumetric shrinkage during cooling and transition from liquid to solid state.

Therefore very unsatisfactory results have been obtained using the basic laboratory method.

With the aim to improve the poor quality of paraffin wax solid grains manufactured with the laboratory method, a second, alternative, more sophisticated procedure has been investigated and experimented, based on the simultaneous application of thermal and mechanical energy, i.e., heat flux and pressure, according to the following main steps:

- the starting point is the raw material in the form of commercially available pellets of SASOL® 0907 paraffin wax;
- inserting the pellets inside a cylindrical mould equipped with embedded electrical resistance heating system and temperature measurements through a K type thermocouple;

- heating the raw material up to a temperature of 110°C, because, as indicated by DSC analysis, this temperature guarantees the complete melting of all linear and branched alkanes present in the SASOL® 0907 paraffin wax.
- holding the temperature of 110°C for the time required for liquefying all the paraffin wax pellets;
- stirring the melt;
- switching off of the electrical resistance heating system;
- application of a pressure of about 10 bar by means of cylindrical piston fitted with a load cell for monitoring the applied compression force;
- slow cooling down to room temperature.

This manufacturing method has been accomplished by the use of special designed equipment, shown in Figure 16, based on the application on a weight lifter of a suitable device for transforming the tensile force component exerted by the crane hook during the lifting phase into a compression force of two opposing plain surfaces. The counter-posed faces of the weight lifter have been adjusted for placing the heated mould and the corresponding piston equipped with the load cell. A load of 300 kg has been set. This value, considering the surface area of the melted paraffin wax, is equivalent to a pressure of about 10 bar, kept constant by means of suitable working of the hydraulic system. The tight coupling tolerances among the mould components have allowed the outgassing of air entrapped inside the paraffin wax during the heating and stirring steps.

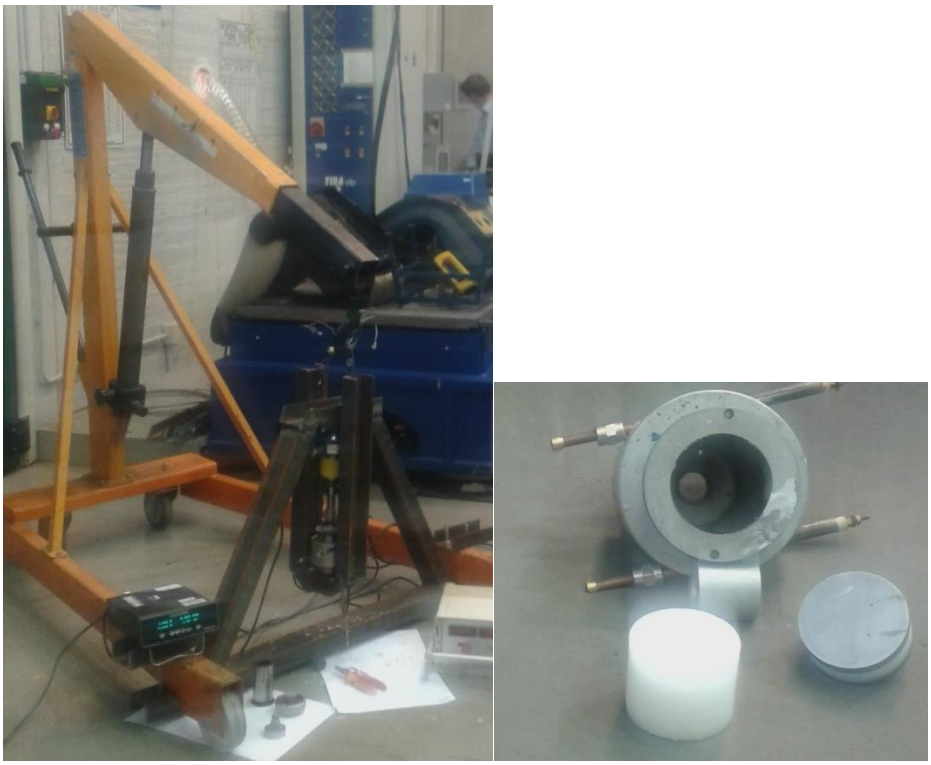

**Figure 16: Metallic box, heated through electrical resistance and pressurized with oleo-dynamic piston used to manufacture the paraffin grain with the die casting method.**

Using this method homogeneous, consistent, solid paraffin wax grains corresponding to configuration 3 in Table 2 have been fabricated as shown in Figure 17.

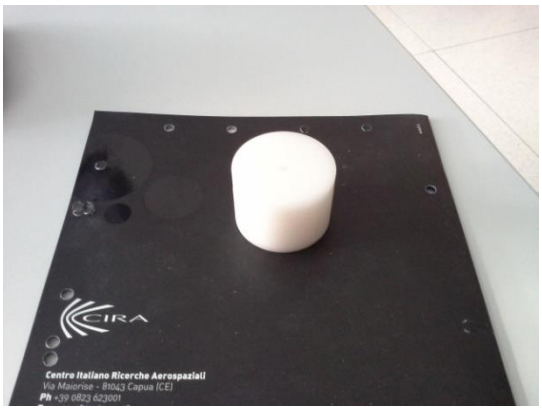


**Figure 17. Paraffin wax solid grain sample manufactured with the die casting method.**

Also in this case, the sample internal microstructure has been microscopically analyzed and the resulting images are shown in Figure 18, in Figure 19 and in Figure 20.

**Figure 18. Fracture surfaces of paraffin wax solid grain samples manufactured with the die casting method.**

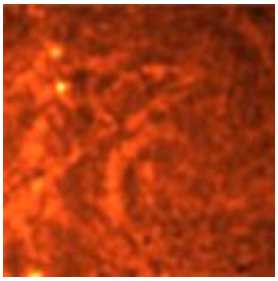

**Figure 19. Optical microscope images of paraffin wax solid grain samples manufactured with the die casting method.**

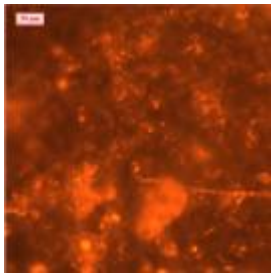

**Figure 20. Optical microscope images obtained in bright field using a magnification of 550x of paraffin wax solid grain samples manufactured with the die casting method**

On one hand traces of pellets not completely liquefied have not been detected and on the other hand no evidence of both surface or internal micro-cracks and micro-porosities of critical size has been noticed.

From the experiments reported in the present investigation only a simultaneous application of external mechanical pressure and an electrical heat flux associated to a bulk temperature inside the liquefying paraffin wax of

at least 110°C is sufficient to avoid the both abovementioned manufacturing problems i.e., non-complete melting of the pellets and presence of micro-porosities.

## IV. Concluding Remarks

Manufacturing processes of microcrystalline paraffin wax solid fuel grains as critical point of design and development of HRE has been addressed and experimentally investigated.
Starting from a scientific literature survey a laboratory, not expensive, repeatable, reproducible, easy to scale method has been selected and optimized based on preliminary, thermal characterization and set up and realization of a special equipment aimed to simultaneous application of heat flux and external mechanical pressure.

Samples fabricated using several methods have been analysed by means of visual inspection and microscopic observations. Specimens manufactured without application of an external pressure show evident internal micro-cracks and micro-porosities due to spontaneous volumetric shrinkages of paraffin wax during cooling and transition from liquid to solid state. Instead samples fabricated with a die casting procedure using the abovementioned special equipment, exerting during cooling a pressure of about 10 bar, appear satisfactory, homogenous, consistent and free from critical size flaws and defects.



## Acknowledgements

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