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Activated and Nano-sized Aluminum as Metal Fuels in Solid Rocket Propellants

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Abstract

The present work describes a methodology for the production of mechanically activated aluminum powders with tailored SSA and reactivity. The obtained specific surface area ranges from about 4 m^2/g up to 90 m^2/g. All the activated powders have been characterized and compared to a nanometric and a standard micrometric aluminum, while three activated ingredients have been selected to be tested in standard solid rocket propellants. The proposed materials exhibit a reactivity and a metal content similar to nanometric Al particles. In solid rocket propellants, the activated powders guarantee an improved burning rate with only a slight increment of the pressure exponent.

1. Introduction

The use of metal powders for the performance enhancement in energetic systems is a strategy implemented from decades. The recent demand for performance increment, e.g. in the solid propulsion field, pushed the research towards the study of innovative materials such as nano-sized aluminum (nAl) and activated aluminum (ActAl). Feasibility studies of nanometric aluminum synthesis ground their roots in the ‘40s, while its large-scale production started around 1990 [1]. This powder offers an excellent reactivity promoted by the reduced size and, consequently, by the high specific surface area (from 6.9 m^2/g to 11.9 m^2/g for commercial coated and uncoated 100 nm powders [2]). However, despite the advantages related to its reactivity, this material is characterized by a relatively low metal content (around 90% w.t. for a high-quality, uncoated 100 nm powder [2]), a moderately high cost, safety concerns and difficult handling. Activated aluminum is an alternative, high-reactive material obtained through a proper activation process. Activation processes can be classified in three categories according to the selected approach.

- Mechanical activation (MA): in this case, the virgin powder and, if present, the additive, are processed by a mechanical treatment. One of the most used approach is ball milling, in which the powder is grinded through a low or a high-energy mill [3],[4].
- Chemical activation (CA): this process consists in treating chemically the powder, by using different ingredients. Results depend on the activation substance and can vary from a pitting corrosion of particles to a weakening of the external oxide layer passing through a “simple” coating of the powder[5],[6].
- Mechano-chemical activation (MCA): in this case, two or more materials are processed together to obtain new chemical species (e.g. intermetallic substances). Generally speaking, a process can be defined mechano-chemical also when a structure change occurs during the process [7].

Among the described techniques, one of the most promising (and one of the most used) is mechanical activation. Firstly introduced in the late ‘60s, the mechanical activation process potentially allows the production of *ad hoc* materials with tailored characteristics making these family of ingredients particularly appealing in several fields [8]. The “new” powders are characterized by a higher reactivity with respect to conventional micrometric ingredients keeping at the same time some of their most interesting advantages (e.g. high metal content, intrinsic safety and relatively low toxicity). To achieve the reactivity of nAl, standard µAl has been activated with several substances such as metal oxides [9][10], polymers [11][12], and metals[13]. Processing of sole aluminum powders resulted in limited reactivity [14], in the production of nanoparticles [15] [16], or in a significant increment of the milling time and of the process complexity [16][17]. The possibility to obtain micrometric powders characterized by a high specific surface area keeping a micrometric scale is particularly interesting from the industrial viewpoint. In this paper a fast high energy mechanical activation (HEMA) method for the production of high specific surface area ActAl powders is presented and discussed in relation to their application in standard AP/HTPB-based solid rocket propellants.

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2. Materials and experimental details

2.1 Powder production and characterization

A propulsion grade 30 µm aluminum powder from ALPOCO Ltd has been selected as starting material. The powder has been processed using a standard planetary mill (RETSCH PM 100) in a 125ml stainless steel vessel using a bimodal distribution of stainless steel balls. The selected BPR was 20:1. The chosen process control agent (PCA) was acetone. The process intensity has been varied changing the rotation speed (from 500 rpm to 600 rpm) and the milling time (from 25 min to 100 min). Processing time higher than 100 minutes were not considered for this experimental campaign. Three powder families, characterized by different activation intensities (from A to C), were produced varying the amount of PCA.

2.2 Powder production and characterization

The produced powders have been characterized in terms of specific surface area, qualitative morphology, metal content and non-isothermal oxidation at low heating rate. SSAs were determined at 77 K by a Micromeritics Tristar 3000 analyzer. Qualitative morphology has been evaluated by SEM images recorded through a JEOL JSM-7600F thermal field scanning electron microscope at 5 kV. Non-isothermal oxidation tests were carried out in air (150 ml/min) at 10 °C/min from ambient temperature to 1050 °C with a SEIKO EXSTAR 6200 TG/DTA machine. The methodology to evaluate the reactivity parameters from TG curves is reported in figure 1 and exploits the variables suggested in [21].

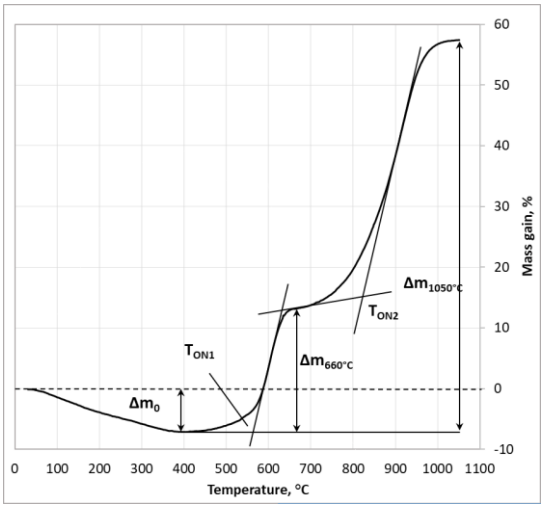

Figure 1: Identification of the reactivity parameters from TG curves

The powder metal content was evaluated by using the same volumetric technique described in [2].

2.3 Propellant manufacturing and testing

Among the produced materials, three activated powders have been selected on the basis of their characteristics and tested in solid rocket propellant (SP) formulations. The target was to compare the combustion behavior of SPs containing ActAl , nAl and µAl powders. Propellants, having the standard formulations AP/Metal/HTPB 68/18/14, were manufactured using a Resodyn LabRAM I resonant acoustic mixer following a standard proprietary procedure. After curing, propellants were cut in samples of 4mm x 4mm x 30mm and laterally inhibited to prevent the flame propagation on the lateral surface. Burning tests were carried out in nitrogen environment in a stainless steel strand burner at different pressures. During the test, the pressure was kept constant through a set of electrovalves controlled by an analog regulator. Combustions have been recorded using a standard video camera and then analyzed by a proprietary digital technique in order to compute the average burning rate. At least three good tests per pressure have been recorded and post-processed.

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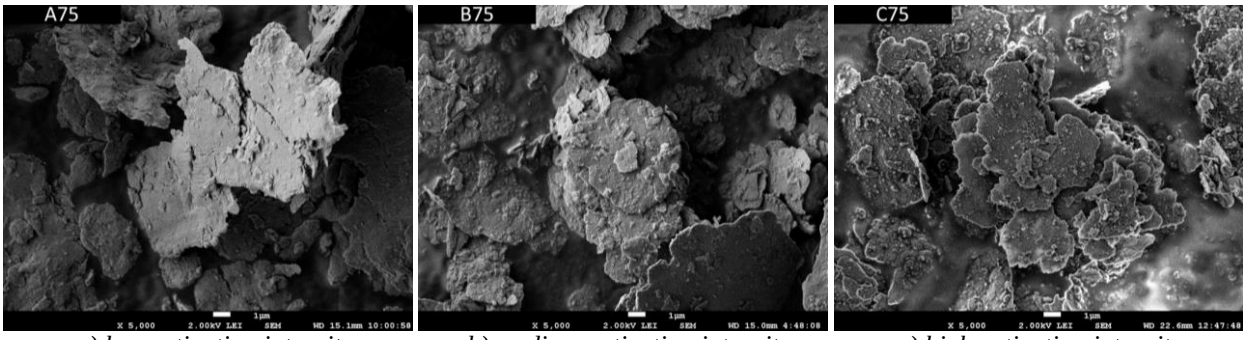
3. Results and discussion

3.1 Powder testing

Table 1 shows the specific surface area and the metal content of the produced ActAl powders. With respect to the virgin material, these powders feature a significant increment of the SSA and a reduced metal content. This effect is a combination between activation intensity and volume of the PCA. A progressive enhancement of the specific surface area and the consequent reduction of the metal content can be observed increasing the activation intensity. This behavior can be observed looking at the samples (A75, B75, C75), (A100, B100, C100) and (A125, B125). All these materials have been produced using the same amount of PCA and different activation intensities.



a) low activation intensity *b) medium activation intensity* *c) high activation intensity*

Figure 2: SEM images at 5000X evidencing the effect of process intensity on powder morphology.

This parameter influences also the morphology of the powders. A high activation intensity, in fact, causes the formation of small objects on particle surface which tends to disappear reducing the milling time and/or the milling speed. Moreover, highly activated particles are thinner than low activated granules as confirmed by the charged spot observable in Figure 2c and absent in Figure 2a. The effect of the PCA volume on particle morphology, SSA and C_{Al} is more difficult to understand. This parameter is responsible for the phenomena occurring during the milling process and plays a key role in the modification of the virgin powder characteristics. In particular, the maximum SSA can be obtained for PCA volume between 7.5 ml and 10.0 ml (see Table 1). PCA volume lower than 7.5 ml causes only smaller values of SSA and the formation of dusty and thick particles. Volume of acetone higher than 10 ml induces important changes in the milling process causing a significant reduction of the specific surface area and substantial changes in the powder morphology: from thick and dusty particles to large and thin flake shape granules (Figure 3).

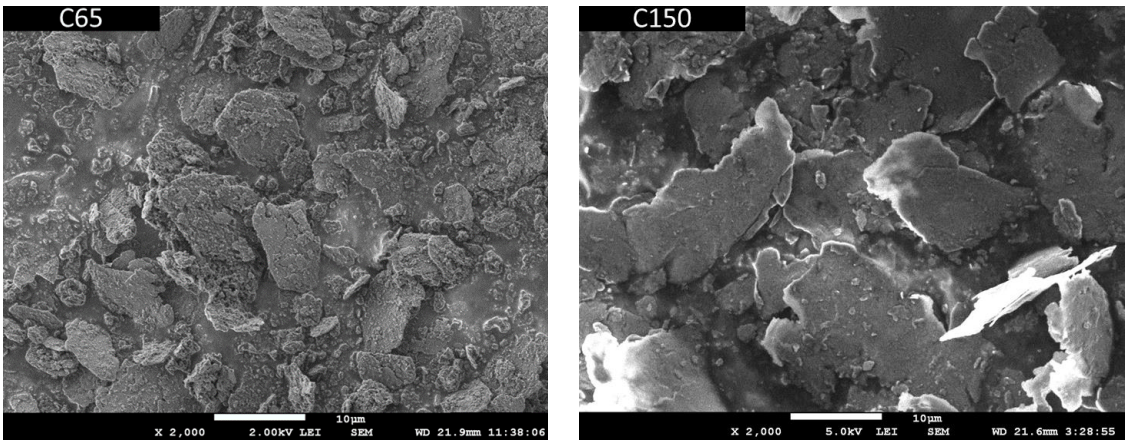


Figure 3: SEM images at 2000X evidencing the effect of PCA volume on particles activated at high intensity.

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Table 1: Metal content and specific surface area of ActAl powders. For comparison, the virgin material and a reference 100 nm nanometric aluminium coated with stearic acid (L-Alex) have been added for comparison (data are taken from [14]). Errors are computed considering a t-students distribution with a confidence level of 95%.

Powder Label	Activation intensity	PCA Volume, ml	SSA, m^2/g	C_{Al}, %
µAl30 (baseline)	No Activation	N. Av.	< 0.1	99.1 ± 0.3
nAl (LAlex-100)	No Activation	N. Av.	10.5 ± 0.1	89.4 ± 1.2
A75	Low	7.5	13.3 ± 0.1	83.1 ± 1.6
A100	Low	10.0	17.4 ± 0.1	85.0 ± 2.4
A125	Low	12.5	4.9 ± 0.1	89.4 ± 2.0
B75	Medium	7.5	36.4 ± 0.1	75.6 ± 0.8
B100	Medium	10.0	21.8 ± 0.1	83.1 ± 1.6
C50	High	5.0	51.8 ± 0.1	74.6 ± 2.3
C65	High	6.5	67.6 ± 0.1	62.8 ± 0.9
C75	High	7.5	87.6 ± 0.1	63.0 ± 1.2
C85	High	8.5	77.2 ± 0.1	55.8 ± 0.8
C100	High	10.0	76.9 ± 0.1	57.8 ± 0.7
C125	High	12.5	31.6 ± 0.1	81.5 ± 0.4
C150	High	15.0	10.4 ± 0.1	84.2 ± 1.3

The proper variation of milling time, milling speed and PCA volume allowed a certain control of both SSA and C_{Al}. As reported in table 1 and figure 4, the production method can be applied for the manufacturing of powders having a SSA ranging from about 5 m^2/g to about 90 m^2/g covering also the typical range of commercial nanometric aluminum (see the red circle in figure 4). The methodology is particularly appealing for the production of samples with a SSA higher than 25 m^2/g. For the proposed activated powders, the C_{Al} depends linearly from the SSA, while, for nanometric powders, the reduction of the metal content goes with the square of the SSA (see figure 4). This means that the powders produced via HEMA and showing a specific surface area greater than 25 m^2/g feature a higher metal content with respect to a corresponding nAl having the same SSA. The reader should notice that the green line sketched in figure4 has been obtained considering a spherical particle having an oxide layer 3 nm thick [2]. .

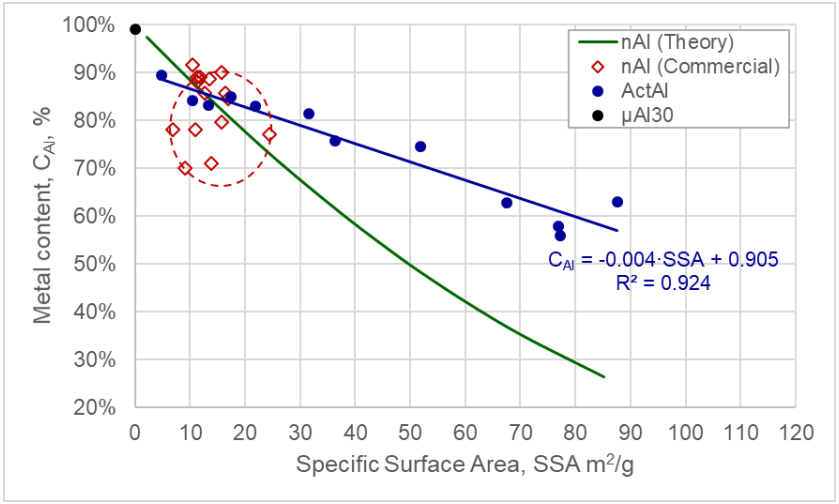

Figure 4: Relation between SSA and C_{Al} for ActAl powders. Values for commercial nAl are added for comparison and take into account both coated and uncoated powders with a nominal diameter from 40 nm to 100 nm [18], [19], [20].

Effects of the PCA volume and of the activation intensity can be detected also on the final powder reactivity parameters reported in table 2. All the powders feature a typical two-step reaction with a more or less marked desorption of gas or PCA before the first oxidation peak (Δm_0). As reported in table 2 and under the investigated conditions, an enhanced activation intensity is responsible for the anticipation of both the first and the second oxidation reaction. On the contrary, the effect of the PCA volume on the two oxidation onsets is quite contradictory and it is not possible to detect

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a clear trend. The effect of the PCA is more evident on the mass change. Increasing the amount of acetone, Δm_0 decreases progressively independently on the activation intensity. According to table 2, the PCA causes a progressive and significant enhancement of the mass gain at 660 °C ($\Delta m_{660°C}$) and at the end of the test ($\Delta m_{1050°C}$) confirming the enhanced reactivity. The maximum values are reached using a volume of PCA between 7.5 ml and 10 ml. This last value represent a sort of limit at which the activation process changes causing an important reduction of the adsorbed acetone, but also a drop of the first mass gain. This behavior is probably related to the possibility of particles to move inside the activation jar and confirms what observed studying the SSA, the metal content and the morphology of the powders.

Table 2: Reactivity parameters for the produced activated powders (TG, air, 10 °C/min). The baseline and a 100 nm nanometric aluminium powder coated with stearic acid (L-Alex) have been added for comparison purposes (data are taken from [14]). The confidence interval for the two runs is defined by the standard deviation.

Powder Label	T_{ON1}, °C	T_{ON2}, °C	Δm_0, %	$\Delta m_{660°C}$, %	$\Delta m_{1050°C}$, %
µAl30 (baseline) [a]	578.6	907.0	-0.0	0.5	7.6
nAl (LAlex-100) [a]	568.0	747.0	-2.9	31.9	73.2
A75	574.2 ± 2.2	872.6 ± 0.3	-1.7 ± 0.1	22.5 ± 0.1	68.4 ± 1.5
A100	567.7 ± 0.9	884.5 ± 1.9	-2.1 ± 0.1	12.1 ± 0.1	67.1 ± 0.2
A125	574.2 ± 0.9	880.0 ± 3.2	-0.6 ± 0.0	12.9 ± 0.0	70.1 ± 0.0
B75 [a]	563.5	817.9	-7.1	21.8	69.5
B100 [a]	566.5	832.6	-3.6	21.5	73.8
C50	544.7 ± 1.7	890.6 ± 10.8	-11.0 ± 0.1	12.7 ± 0.2	46.4 ± 0.2
C65	559.6 ±1.6	840.6 ± 0.7	-14.5 ± 0.3	20.7 ± 0.4	61.0 ± 1.2
C75	558.5 ± 0.7	763.9 ± 3.7	-12.6 ± 0.5	30.0 ± 0.6	65.9 ± 4.7
C85	563.1 ± 1.0	769.4 ± 1.6	-14.1 ± 0.8	31.0 ± 0.1	63.1 ± 0.1
C100	562.8 ± 2.2	774.8 ± 3.4	-16.9 ± 0.2	30.7 ± 0.5	61.7 ± 0.9
C125	568.0 ± 0.4	826.3 ± 1.4	-3.2 ± 0.2	24.0 ± 0.0	72.1 ± 0.9
C150	567.4 ± 3.1	848.2 ± 1.2	-1.5 ± 0.7	20.9 ± 0.3	75.2 ± 0.4

[a] only one test available

All the produced samples exhibit a substantial enhanced reactivity and a peculiar morphology with respect to the virgin micrometric material. Looking at the reference nAl powder, only a reduced number of activated aluminum exhibits similar characteristics, especially looking at the $\Delta m_{660°C}$ parameter. On the basis of these similarities and looking at all the powder characteristics, three samples have been selected for the experimentation in solid propellants. The powder C150 was selected due to the similarities with the reference nAl in terms of SSA and metal content and final mass gain. Moreover, this powder exhibits the lowest adsorption of PCA and a good first mass gain. The sample C75 has been chosen because it is the sample showing the highest specific surface area, while the powder A75 represented the best compromise between reactivity and morphology parameters.

3.2 Propellant combustion

The formulations of the tested solid propellants are reported in table 3. Both total and partial replacement of standard micrometric aluminium have been assessed.

Table 3: Tested solid rocket propellant formulations

Propellant Label	Binder, %	AP_{coarse} %	AP_{fine} %	µAl30, %	L-nAl, %	ActAl, %
P-µAl30	14	58	10	18	0	0
P-nAl	14	58	10	0	18	0
P-nAl_3	14	58	10	15	3	0
P-A75	14	58	10	0	0	18 (A75)
P-C75	14	58	10	0	0	18 (C75)
P-C75_3	14	58	10	15	0	3 (C75)
P-C150	14	58	10	0	0	18 (C150)

DOI: 10.13009/EUCASS2019-791

The burning rates of SPs loaded with a single metal powder are reported in figure 5. The replacement of baseline ingredient with highly reactive powders brings to a significant increment of the burning rate. Under the investigated conditions, the reference nanometric aluminum guarantees a r_b improvement between 64% and 80% with respect to the baseline. The use of activated powders can reproduce the behavior of the nanosized aluminum (sample P-A75) or can guarantee a higher burning rate with respect to the nanoaluminuzed formulation (about +6 % for the propellants P-C75 and P-C150). The pressure sensitivity tends to rise increasing the specific surface area of the metal powder. This means that all the tested formulations are characterized by a higher pressure exponent with respect to the baseline; from 0.46 to 0.49 for the nanometric aluminum and up to 0.55 when the powder C75 is used.

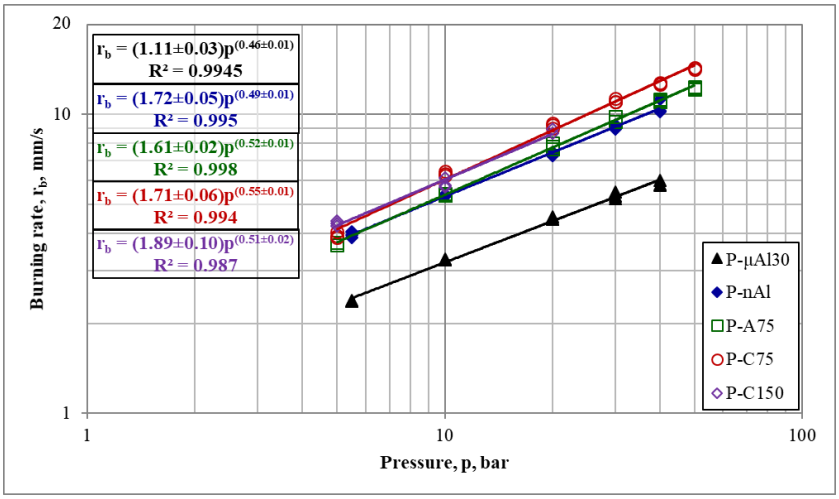

Figure 5: Vieille's law of the propellants loading a single aluminium powder. A propellant loaded with a standard micrometric Al (P-µAl30) and a propellant loaded with a 100 nm aluminium coated with stearic acid (P-nAl) have been added for comparison.

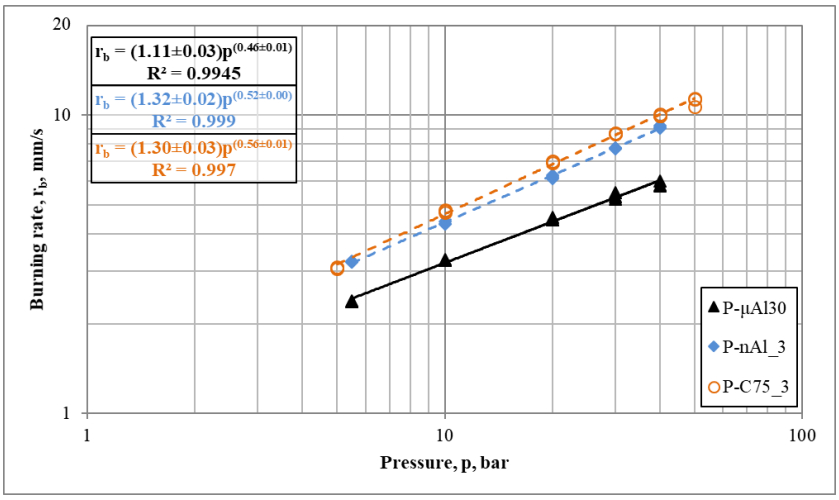

Figure 6: Vieille's law of the propellants loading Al powder mixtures.

Significant increment of the burning rate with respect to the baseline can be obtained also with a partial replacement of the standard micrometric powder. Using a mixture µAl/nAl 15%/3% it is possible to improve the burning rate up to the 55 % at 40 bar. Better results has been obtained by means of the same mixture containing the powder C75, which is capable to enhance the r_b up to +70 % at 40 bar. The pressure exponent is higher when metal mixtures are used. However, the values remain sufficiently low (0.52 and 0.56 for the propellant P-nAl_3 and P-C75_3 respectively).

DOI: 10.13009/EUCASS2019-791

3.3 Discussion

The possibility to produce high SSA mechanically activated powders with an enhanced reactivity has been demonstrated. However, the opportunity to use these powders, but also nanometric aluminum, in standard solid rocket propellants should be discussed considering both production and performance issues. From a propulsive point of view, the use of metal fuels with a relatively low reactivity (e.g. µAl) is responsible for a consistent reduction of the ideal specific impulse, due to the presence of condensed combustion products (CCPs) expanding through the gas-dynamic nozzle. The presence of burning droplets of molten Al (agglomerates) in the solid rocket core flow causes a detriment of the ideal specific impulse ranging from 1% to 3% [22]. Size and mass fraction of CCPs have been found to be relevant parameters for performance detriments due to thermal and velocity lag of condensed particles [22][23]. If the increment of metal powder reactivity is the strategy selected for the reduction of CCP size, and thus for the improvement of the delivered specific impulse, it is necessary to guarantee at least the specific impulse defined by the red line in figure 7. Lower values will bring to a reduction of the delivered performance with respect to a standard powder. For this reason, the complete replacement of µAl with some nAl or with the proposed activated powders should be carefully evaluated due to the relatively low metal content (ideal specific impulse reduction). Looking at the combustion behavior and at figure 7, a partial substitution of the standard powder is a better solution than a total replacement. A limited amount of nanometric or activated powders does not represent a problem from the production viewpoint and guarantees a good enhancement of propellant burning rate [2][14][24][25]. The selection between a nanometric and an activated powder of the type considered in this work (which is still micrometric) will be then mainly related to the specific application and to health and safety concerns.

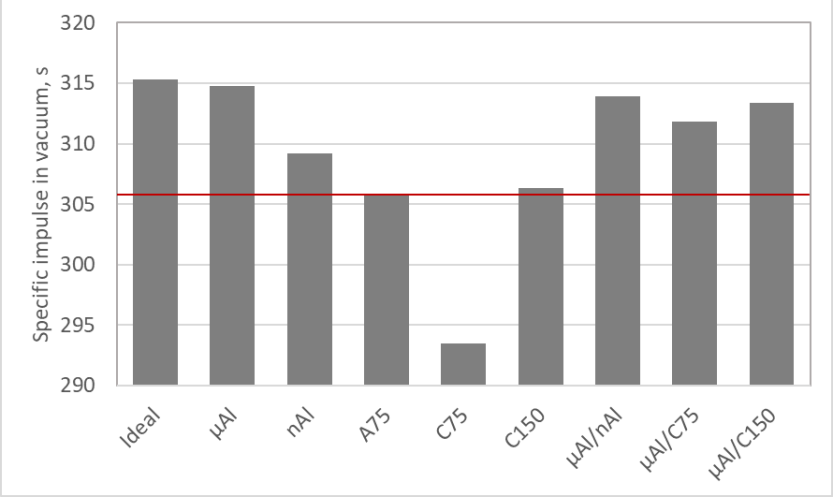

Figure 7: Specific impulse in vacuum for aluminized propellants loading different metal fuel. Thermochemical computations were carried out with the CEA code in shifting equilibrium conditions considering a combustion chamber pressure of 70 bar and an area ratio of 40. A standard formulation (HTPB/AP/Metal – 14%/68%/18%) has been considered.

4. Conclusions

The present work describes a viable methodology for the production of mechanically activated aluminum powders with tailored SSA and reactivity as possible alternative to nanometric aluminum. Several powders have been produced and characterized starting from a propulsion grade micrometric Al with nominal diameter of 30 µm. Activated powders have been characterized and compared to a reference (target) nanometric Al ingredient. A selection of ActAl powders have been tested in solid rocket propellants. A proper variation of milling speed, processing time and PCA volume allowed a certain control of both specific surface area and metal content of ActAl samples. For the produced activated materials and in the investigated SSA range, the two parameters are related by a linear law instead of the quadratic one typical for the spherical nanoparticles. This peculiarity is probably imputable to the morphology of the activated powders (composed by flake shape particles) and it represents an interesting advantage when ingredients with a SSA greater than about 25 m^2/g have to be produced. In the range from 8 m^2/g to 25 m^2/g, the activated materials can reproduce the typical metal content values of coated and uncoated propulsion grade nAl powders. Tailoring the milling process, reactivity parameters of ActAl samples become similar to those shown by the target nAl. However, ActAl

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samples showing high values of SSA are characterized by a strong adsorption of acetone which can be removed by a proper drying cycle.

When embedded in solid rocket propellants, the selected activated powders can improve the burning rate much more than the reference nAl with only a minor increment of the Vieille's law pressure exponent. This is valid for both total or partial replacement of the standard metal. Despite the increased reactivity, a total replacement of µAl with nAl or ActAl cannot guarantee the enhancement of the delivered specific impulse due to their relatively low metal content. On the contrary, the adoption of nAl and ActAl in SPs is interesting when a partial replacement of standard ingredients is considered. In this case, the enhanced reactivity of the two powders can efficiently reduce the size of CCPs keeping a high theoretical specific impulse. Focusing the attention on this last strategy, the selection of one of the two material families is simply related to the specific application and to safety and health concerns.

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