New High Energy Mechanical Activation 2nd gen (NHEMA2). Scale up and perspectives of the new technique for activated thermite production

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Abstract

This work aims to compare two generations of high-energy mechanical activation techniques based on resonance: NHEMA1 and NHEMA2. Due to the renewed interest in tailored energetic materials, activated Fe_2O_3/Al thermite has been selected as "case study" to describe the potentialities of the new process and the differences with respect to the NHEMA1. Four mechanically activated thermites have been produced by NHEMA2 and characterized in terms of metal content and ignition temperature. Results have been compared with a baseline material manufactured through NHEMA1. Production capabilities (existing and future perspectives) have been presented and discussed.

Keywords: *Ball Milling, Mechanical Activation, NHEMA2, Activated thermites, Scale-up*

1. Introduction

High-energy ball milling (HEBM) is a "batch production" technique employed in several fields (e.g. propulsion, pyrotechnics, bioengineering, batteries, hydrogen generation, etc.) to synthesize specialized materials with tailored characteristics (Benjamin, 1970; Koch & Whittenberger, 1996; Musicco et al., 2025; Zhang, 2004). Introduced in the late '60s, this methodology has been recently indicated as a valuable support to green chemistry, also thanks to some peculiarities such as the low processing temperature and pressure, and the possibility of avoiding organic solvents (Pantoya et al., 2009). HEBM processes rely on proper machines called "ball mills", mainly differing in the specific motion induced and in the vessel shape (and size). The most well-known lab-scale, high-energy mills are the planetary mills, the attritor mills, and the shaker mills (Suryanarayana, 2001). All these machines can be efficiently employed in HEBM synthesis with a typical process duration between 30 minutes (for the most recent devices) and a few hours (Suryanarayana, 2001). Despite the interesting features, the scale-up of the technology is not simple, and it has been hindered by several issues, like energy consumption, temperature control difficulties, and milling media dynamic variation. On the contrary, low-energy ball milling (LEBM) is already available at an industrial level. Drum mills and 3d mills are two examples of low-energy devices used for LEBM. Despite the long processing time (several hours), they are characterized by significant production capabilities and are widely employed at an industrial level (e.g, for the production of low-cost metal powders) (Feng et al., 2023). Unfortunately, the LEBM cannot always be applied, especially when the production of high-performance materials is requested (Al Bacha et al., 2020). In this respect, some years ago, a new HEBM technique based on a resonance approach (the NHEMA1) was introduced to solve some of the problems encountered using conventional techniques. The NHEMA1 proved to be faster and more versatile with respect to standard procedures, but with some limitations in terms of machine price and procedure

versatility. The technique has been significantly modified starting from 2022. The new process (the NHEMA2) employs a new and less expensive device capable of better transferring the energy to the powders, thus leading to a more efficient and faster material production (Dossi et al. 2023; Dossi et al., 2024). The device can be rapidly modified and adapted to optimize the process on the basis of the requested final results. Finally, the wide regulation of parameters that were not controllable with the NHEMA1, makes the NHEMA2 more versatile and efficient.

This work compares NHEMA1 with NHEMA2 processes. Mechanically activated Al/Fe_2O_3 thermites are considered a case study to assess production capability, versatility, and performance of materials obtained through the two techniques. Mechanically activated thermites are analyzed in terms of ignition temperature and active metal content as an indication of HEBM aggressivity. One of the most interesting features of the HEBM is the possibility of tailoring the characteristics of the produced material (Dudina & Bokhonov, 2022). This capability is particularly interesting in many applications, including satellite demise (Finazzi et al., 2024) where the tailoring of ignition temperature plays a very important role. In this respect, NHEMA2 offers improved capabilities that will be shown and discussed

2. Materials and methods

2.1 Materials

Propulsion-grade aluminum powder with a declared d(0.5) of 30 μm and a purity above 99.5 % from ALPOCO LTD was activated with micrometric iron oxide (98 % purity) from Thermo Fisher. The mixture has been processed by NHEMA2 methodology in air (Dossi et al. 2023; Dossi et al., 2024). The main production parameters are reported in Table 1. The specific values have been selected to change the activation intensity, thus tailoring the ignition properties.

Table 1. Production details of tested thermites.

Label	Method	Overall processing time, min	BPR	Formulation, wt. %	
				Al	Fe_2O_3
AT2_1	NHEMA2	6	20:1	26	74
AT2_2	NHEMA2	13	12.5:1	26	74
AT2_3	NHEMA2	7	12.5:1	26	74
AT2_4	NHEMA2	3	10:1	26	74
AT1_ref	NHEMA1	19	20:1	26	74

For comparison purposes, the best thermite activated by NHEMA1 has been considered as baseline material (Agostinelli et al., 2020; Dossi et al., 2018). After production, the thermites were stored at ambient temperature in a closed polypropylene vessel. Silica gel was used as a desiccant agent to lower the moisture in the storage box.

2.2 Testing procedures and equipment

The samples have been tested in terms of active metal content and ignition temperature at heating rates ranging from 30 °C/s to 450 °C/s. All the tests presented in this work were performed in accordance with internal protocols defined within the framework of the ISO9001 certification.

2.2.1 Metal content tests

Metal content tests were carried out using the displacement method described in (Chen et al., 2010) with a 5 wt.% NaOH aqueous solution. Approximately 200 mg of powder was weighed using a microbalance and inserted into a paper pot. The sample was then placed inside an "upside down" graduated cylinder, and water displacement was evaluated. The metal content was computed by converting the volume of evolved hydrogen, considering the following reaction

$$Al + 3H_2O \rightarrow Al(OH)_3 + \frac{3}{2}H_2$$

A minimum of three tests per sample were carried out.

2.2.2 Ignition tests

Ignition tests were carried out in air using a hot-wire setup composed of a NiCr resistive ribbon, a 600 W power supply, and a calibrated one-color optical pyrometer. The ribbon was used to heat the sample at the desired heating rate by the Joule effect, while the optical pyrometer measured the ribbon temperature near the sample. The experimental procedure was composed of the following steps:

- Positioning of the sample on the heating ribbon
- Pyrometer position adjustment to target the proper portion of the heating element
- Activation of the power supply, ensuring the proper heating rate

The temperature profile was recorded and then analyzed by an Octave script to detect the ignition temperature.

2.2.3 Estimation of the production rate

Production rate of both NHEMA1 and NHEMA2 has been estimated based on the current maximum production capability per batch. The baseline material and its performance have been considered as a reference. Automation was not contemplated; thus, a typical time necessary for material extraction was added to the standard processing time. The computation relied on the hypothesis that a single operator works on the machine for about 1700 hours per year. For both techniques, the limit of the current pilot technology was considered. The computation of the maximum production was based on the potential production enhancement obtainable by acting on the activation device, on the vessel size, and on the process.

3. Results and discussion

The effect of activation intensity is clearly visible from the powder Al content reported in Figure 1. More aggressive processes caused by prolonged activation time or by a high ball-to-powder mass ratio led to a consistent reduction of the metal content. It is worth noting that the powder AT2_1, processed for 6 minutes by NHEMA2 (BPR of 20:1), features the same metal content of the sample AT1_ref produced in 20 minutes by NHEMA1, testifying to the superior energy transfer guaranteed by the new generation process.

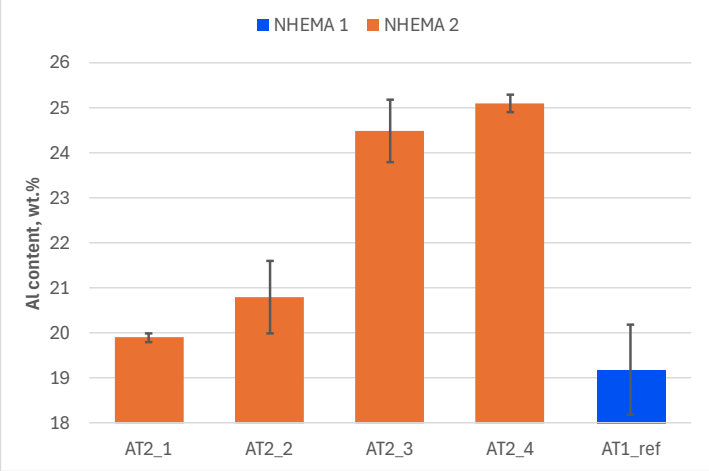

Figure 1. Thermite metal content.

Despite the similar metal content, the sample AT2_1 ignites 150 °C before the baseline, confirming the higher efficacy of the NHEMA2 (Figure 2B). The effect of the activation intensity is clearly observable when comparing the metal content and the ignition temperature of the AT2 sample family.

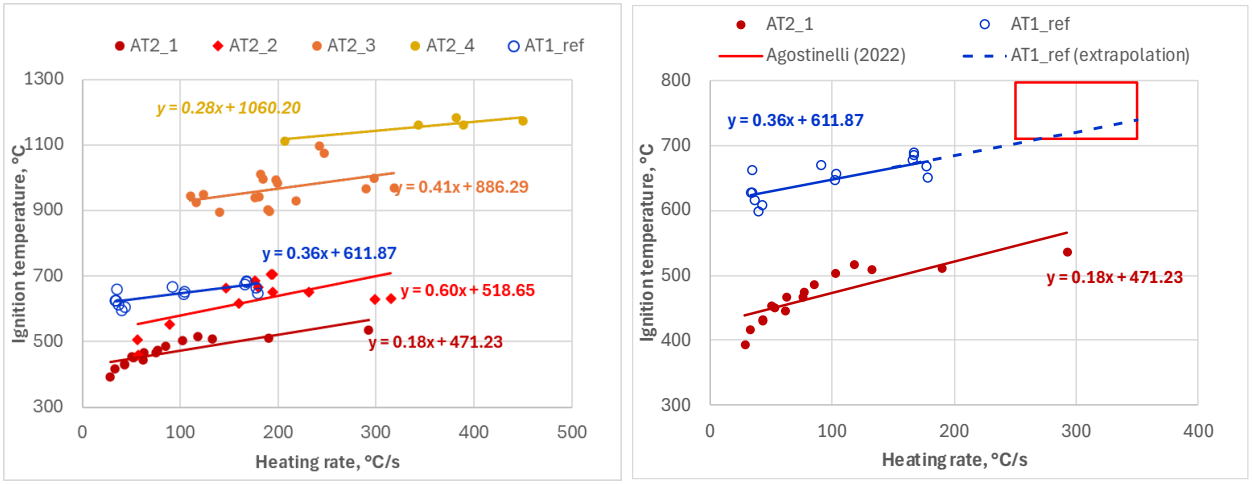

Figure 2. Ignition temperature vs heating rate. (**A**): all samples (**B**) direct comparison between NHEMA1 and NHEMA2

A progressive reduction of both active aluminum content and ignition temperature can be observed when the process intensity is increased by incrementing the BPR and/or by increasing the processing time (Figure

1 and Figure 2A). It is worth noting that the NHEMA2 can be tailored to promote a relatively precise variation of the ignition temperature. The four thermites produced by NHEMA2 exhibit an ignition temperature between 400 °C and 1180 °C. From this viewpoint, it is necessary to consider that HEBM is a complex process involving numerous independent parameters. Their variation is responsible for the change of the final material characteristics, including homogeneity, but their combined effects are neither linear nor easily predictable. Thus, similar results can be achieved by changing different sets of processing variables. In this respect, both the techniques offer a remarkable versatility, but the new generation guarantees the control of more processing parameters.

Current and predicted production capabilities for mechanically Al/Fe_2O_3 thermites of NHEMA1 and NHEMA2 are reported in Figure 3. The introduction of the NHEMA2 significantly improved the production rate of activated thermites by more than ten times, keeping the same material performance. Existing NHEMA2 pilot plant can produce up to 200 kg of activated thermites per year, overcoming the maximum limit of 145 kg estimated for the NHEMA1. However, the maximum limit of the NHEMA2 pilot machine is expected to be higher than 1 ton. Better results can be obtained by introducing automated processes.

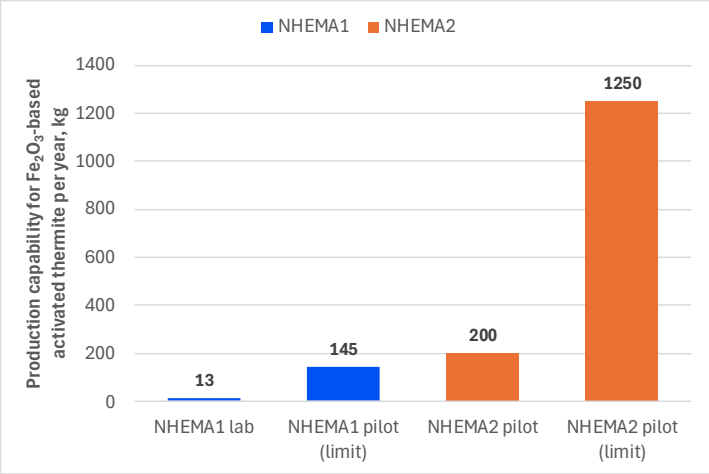

Figure 3. Current and maximum production capabilities of NHEMA1 and NHEMA2 pilot plants.

4. Conclusions

The work compared and discussed two innovative mechanical activation techniques based on resonance, NHEMA1 and NHEMA2, on the basis of production capabilities (existing and expected), versatility, and material performance. Fe_2O_3/Al-based thermites were selected as an interesting case study due to the recent interest in these material family. Five powders, one produced by NHEMA1, have been manufactured and characterized. Metal content and ignition temperature-to-heating rate dependence were measured and critically discussed on the basis of the specific production procedure. A basic discussion on scale-up possibilities of NHEMA1 and NHEMA2 was also provided.

NHEMA2 demonstrated a better scalability than NHEMA1 thanks to the number of selectable parameters and to the higher amount of energy transferred to the sample resulting in a faster and more productive technique. Samples activated by NHEMA2 resulted in better performance (lower ignition temperature and

higher metal content) despite the lower processing time. The temperature ignition tailoring by changing processing parameter has been demonstrated.

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