



Microwave ignition of the combustion synthesis of aluminides and field-related effects



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ABSTRACT

Combustion synthesis of aluminide intermetallics starting from reactive powders mixtures comprising a ferromagnetic component (Fe, Co and Ni) has been performed by igniting the exothermic reactions in a microwave single mode applicator, in order to investigate possible effects related to the positioning of the reactive sample into the region of predominant electric or magnetic field.

In addition to an increase of the reproducibility of the experiments and the possibility to avoid electric arcs generation, the ignition of the combustion reactions in predominant magnetic field allowed to significantly reduce the ignition times, with a global reduction of the power required to synthesize the intermetallics. This implies that the amount of the reactive volume under ignition conditions is reduced proportionally, thus moving from thermal explosion towards Self-propagating High-temperature Synthesis mode of combustion.

Moreover, in the case of Fe–Al system, the disposition of the load into the region of predominant magnetic field increased the yield of the desired intermetallic compound, to the detriment of α -Fe(Al) solid solution co-product. Furthermore, the possibility to continue to furnish energy to the reactive Fe + Al sample after combustion synthesis ignition has been proven for both configurations. Results show that in case of exposure to the predominant magnetic field, the sample cooling rate resulted lower, hence the permanence at high temperatures after synthesis could be proficiently exploited to increase adhesion to an underlying substrate, in case combustion synthesis technique is used to obtain intermetallic-based coatings.

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1. Introduction

Aluminide intermetallic compounds and phases are currently being investigated for their use in high temperature structural and protective applications, mainly due to their low densities, high melting temperatures and the high aluminium content, which guarantees excellent high temperature oxidation resistance [1].

Combustion synthesis (CS) is widely recognized as an advanced materials processing technique characterized by several advantages over more conventional fabrication procedures [2,3], which are mainly related to its extremely low energy requirements. Indeed, energy input is limited to the ignition step, since the exothermic reaction initiated is able to become self-sustained as a result of the heat produced (and hence the energy released) by the reaction itself.

Among the large number of possible ignition techniques, microwave (MW) energy surely represents an attractive alternative, mainly as a consequence of its characteristic heating mechanism, which is based on the interaction of electromagnetic energy with matter according to its electric, dielectric and magnetic properties. Direct interaction of the microwave energy with absorbing reactive samples allows MW assisted ignition to benefit of several advantages, with respect to conventional thermal ignition, including ignition time savings together with energy efficiency, the latter due to the possibility to limit the heat generation to the sample alone, not involving, for instance, the furnace walls or the hot plate and the eventually employed sample holder [4]. Being the heat generated directly into the sample and not transferred to it by a radiating surface, the volumetric nature of microwave heating, that is directly correlated with the penetration depth of the microwaves into the reactive material [5], constitutes a further peculiarity of microwave ignition with respect for example to more localized ignition sources like a typical electrically heated tungsten coil or even a laser [6]. Moreover, differently from other ignition sources, like mechanical

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impact one [4,7], the use of microwaves allows a non-contact transfer of energy, thus potentially avoiding any contamination, and most importantly their selective nature can be proficiently exploited joining applications, as reported elsewhere [8].

Moreover, ignition of combustion synthesis reactions by means of microwaves at 2.45 GHz is used to achieve complete conversion of reactants [9–11] and to obtain products with unique microstructures [12]. However, despite its doubtless green process intensification character, this synthetic route often lacks of reproducibility due to the electromagnetic field distribution-related ignition mechanisms, which range from the direct heating of the reactants, to their indirect heating by the use of microwave co-absorbers [13] to the possible occurrence of breakdown phenomena [13,14].

In this work, the effects of microwave ignition of CS reactions in regions of predominant electric (E) or magnetic (H) field [15] are investigated for mixtures of aluminium powders with powders of one ferromagnetic metal reactant (namely Fe, Co and Ni), in order to increase the process reproducibility and propose new strategies to optimize the synthesis of high purity aluminide intermetallics, being this latter aspect of paramount importance for several practical applications. Indeed, although the ductility-related drawbacks of some intermetallic compounds still represent a significant hurdle before their full utilization as structural materials could be reached, some proposed strategies (e.g. addition of opportune alloying elements [16] and reduction of the crystallite size to the nanometre range [17]) contributed to partially overcome those limits, enabling their use in several successive processing routes, like for example thermal spray and additive manufacturing [18–20]. Moreover interest towards intermetallic foams is significantly increasing [21,22], and the use of aluminide intermetallics as high temperature protective coatings is yet a well-established practice. However, some of the proposed strategies for the obtainment of intermetallic-coatings like for example hot-dipping [23], mechanical milling [24], pack aluminizing [25] and thermal spray [26] possess several disadvantages including long term high temperature heating steps, together with the need of preliminary mechanical alloying treatments or post deposition annealing ones.

Thus, in this perspective, the possibility to replace the existing thin intermetallic films and coating manufacturing techniques, and generally the intermetallic compounds fabrication strategies, with the here presented combustion synthesis-based approach doubtless represents a stimulating research field, with significant practical implications. Indeed, despite the significantly reduced manufacturing time and cost, associated with combustion synthesis, the use of microwave energy as ignition source can lead to all of the above mentioned benefits, together with the possibility to exploit also the interaction of some reactive precursors with the magnetic field component of the incident microwaves, which can lead to significant heat contributions, helping the synthesis of the desired phases, without the need of post combustion heat treatments.

As a matter of fact it is well known that the microwave power density generated in a volume of the load can be expressed by Ref. [27]:

$$P_d(x, y, z) = \omega \varepsilon_0 \varepsilon''_{\text{eff}} E_{\text{rms}}^2 + \omega \mu_0 \mu''_{\text{eff}} H_{\text{rms}}^2 \quad (1)$$

where P_d = power density in the material (W/m^3), at the position (x, y, z) ; $\omega = 2\pi f$ (Hz); f = frequency of the incident microwaves; $\varepsilon''_{\text{eff}}$ = effective loss factor, including conductivity losses; μ''_{eff} = imaginary part of the effective magnetic permeability; E_{rms} = local (x, y, z) electric field intensity (V/m); H_{rms} = local (x, y, z) magnetic field intensity (A/m). Thus, microwave heating of ferromagnetic powders presents a strong contribution by the H field

interaction with matter, which, in regions of predominant magnetic field, can result significantly higher than the electric field related contribution.

Moreover, electric and magnetic properties affect also the skin depth at 2.45 GHz, which for a given electrical conductivity, results much smaller in case of ferromagnetic powders.

This implies that in the presence of ferromagnetic powders loads, not only the power density, but also the extension of the regions where heat generation occurs are affected, depending on the load electrical and magnetic properties as well as on its arrangement inside the microwave cavity.

2. Experimental

Aluminium powders ($-75 \mu\text{m}$, 99% purity, Sigma Aldrich, Milan, Italy) were mixed to one ferromagnetic powder among:

- Fe ($-45 \mu\text{m}$, 97% purity, Sigma Aldrich, Milan, Italy) in the 60:40 at% ratio (if not otherwise specified)
- Co ($1.6 \mu\text{m}$, 99.8% purity, Alfa Aesar, Karlsruhe, Germany) in the 50:50 at% ratio
- Ni ($\sim 5 \mu\text{m}$, 99.7% purity, Sigma Aldrich, Milan, Italy) in the 50:50 at% ratio.

The selected at% compositions correspond to those characterized by the highest melting point inside the existing fields of the target intermetallics (i.e. the equiatomic FeAl, CoAl and NiAl), according to the corresponding binary phase diagrams.

The larger particle size of Al was selected in order to minimize surface oxidation during handling and since the aluminide combustion synthesis mechanism usually involves reactions between the molten Al and the solid ferromagnetic powder added. Different particle size of the ferromagnetic powders were selected in order to investigate size-related effects, with the larger particles presenting a higher possibility of remaining partially unreacted.

Mixing was performed in an Ar-filled alumina jar for 10 min with alumina balls milling media. X-ray diffraction (XRD, X'Pert PRO, PANalytical, Almelo, The Netherlands, $20 \leq 2\theta \leq 70$, Cu-K α radiation, scanning rate of $0.004^\circ \text{s}^{-1}$ and a step size of 0.02°) was used to confirm that no reaction occurred between the powders after mixing, as well as to investigate the phase formed after combustion reactions. Controlled amount (1 g) of the opportune powders mixture was uniaxially pressed in a cylindrical stainless steel mould at 150 MPa pressure in order to obtain cylindrical pellets of 12 mm diameter and 2–3 mm height. Such reactive samples were then positioned on a low loss refractory support in the regions of predominant E field or predominant H field of a TE₁₀₃ microwave single mode applicator operating at the frequency of 2.45 GHz [10].

Microwave forward power was set to 300 W, which resulted enough to ignite the combustion synthesis in the case of Fe–Al and Co–Al systems, while 1000 W were required for the ignition of the Ni–Al one, as explained later. Microwaves were turned off immediately after combustion synthesis ignition (if not otherwise specified).

In order to perform an accurate and precise comparison between ignition in predominant E and predominant H field, before the ignition of each CS reaction, preliminary impedance matching operations were realized, by means of a shorting plunger and a three-stubs tuner, with the aim to have approximately the same starting value of reflected as well as dissipated power, thus assuring to process the samples with similar initial heat generation conditions.

Surface temperature measurements were performed by means of both an infrared pyrometer (OPT CT LT15F, temperature

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