Study of burning rate characteristics of propellants containing Al–Mg alloy nanopowder

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Abstract
Nanopowder of Al–Mg alloy was synthesized using the method of electrical explosion of thin wires of hypo-eutectic Al–Mg alloy. Thin wires of hypo-eutectic Al–Mg alloy were cut by electrical discharge machining and exploded electrically. The nanopowder obtained is examined using scanning electron microscopy (SEM) and transmission electron microscopy (TEM) for studying the morphology of the particles. TEM and SEM images substantiate the spherical structure of the nanoparticles and the diameter of particles ranges from 20 nm to 95 nm. Solid propellants were prepared by adding the alloy nanopowder as an energetic additive element with bimodal ammonium perchlorate (AP) and Hydroyxl Terminated Polybutadiene (HTPB) propellant formulations. Burning rates of the propellant with nano Al–Mg alloy powder are compared with micro aluminised propellants and propellants with micro Al–Mg powder with the same composition. Mean burn rate of 22.03 mm s\textsuperscript{-1} was obtained at a pressure level of 9 MPa with nano Al–Mg powder and 20.4 mm s\textsuperscript{-1} with micro Al–Mg powder compared to mean burning rate of 10.03 mm s\textsuperscript{-1} at 8 MPa pressure for aluminised propellants. The improved mean burn rate of propellants containing Al–Mg alloy nanoparticles could be accounted for the reduction in the size of the alloy particle and selective melting of eutectic phases along with magnesium. In the process, it gives heat feedback to the high-temperature reaction zone, which results in augmenting further reaction of metal particles.

1. Introduction

Rockets with solid fuels have been in use since the early 13th century. Solid fuels are used for various applications such as sounding rockets, missiles, orbital rockets etc. Solid rockets powered by gun powder were the predecessor of the modern–day solid rockets powered by composite propellants with high thrust and specific impulse ($I_p$). Historical illustrations dated back to early 13th and 14th century depict solid propellant rockets termed as fire arrows. Solid propellants can be broadly classified to single, double and triple based propellants where organic nitrate-based explosives viz nitrocellulose, nitroglycerine, nitroguanidine etc are used and composite propellants where combustible binders such as synthetic rubber, HTPB and oxidizers such as ammonium perchlorate, ammonium nitrate, lithium chloride, potassium perchlorate along with metal powders such as aluminium, magnesium, boron, lithium etc as energetic additives, are used. Jack Parson from Caltech was a pioneer in making castable composite propellants with asphalt as a binder and potassium perchlorate as an oxidiser in 1942 \cite{1}. Metal particles are added as an energetic additive in solid propellants along with oxidiser and binder to improve energy content and specific impulse. Elements like aluminium, boron, magnesium are of
a major area of interest in such energetic application due to higher energy content in the oxidation reaction, low cost and relatively higher abundance in earth’s crust. Aluminium constitutes the major metal particle as an energetic element in solid propellants. Elemental aluminium is highly reactive, it gets oxidised readily and a layer of aluminium oxide (alumina) is formed around [2]. Alumina is highly stable and has a melting point of 2,072 °C. Alumina has a density of 3.98 g cc\(^{-1}\), as compared to the density of aluminium which is 2.7 g cc\(^{-1}\). The high melting denser alumina layer is less permeable to oxygen which results in delayed ignition. Whereas oxide of magnesium is less dense than magnesium and offers low resistance against diffusion of oxidiser [3].

Nanopowder of metals shows higher reaction rate due to the increase in surface area and a higher number of surface dangling bonds compared to the equivalent mass of bigger particles. Jayaraman et al [7] synthesized nanopowder of aluminium by electrical wire-explosion method and used as a metal additive in solid propellants with AP and HTPB [4]. They have reported that the propellants with nano-aluminium powder showed better mean burning rate compared to propellants containing micro-aluminium powder. Gnanaprakash et al [5] studied the effects of nanopowder in matrices and propellant sandwhichs and found that the nano-aluminized matrices exhibit higher burning rates than the non-aluminized ones, due to the enhanced heat feedback available through nano-Al combustion. The sandwich propellants were prepared with fine AP of two different size distributions viz 5 μm and 45 μm. The nano-aluminized sandwiches containing larger fine AP particles(45 μm AP) exhibit enhancement in burning rate. Pure aluminium nanopowder has a large volume fraction of oxides formed naturally at the cost of lowering net energy content [2]. Large agglomerates produced in metal combustion lead to two-phase flow losses which can affect specific impulse substantially [2, 4]. Jayaraman et al [4] also observed that nanopowder of aluminium contains 10%–25% of oxide by weight which leads to a reduction in specific impulse and production of larger agglomerates at higher metal loading leading to two-phase flow losses.

Reactivity of metal powders can be improved by reducing their particle size, surface activation and by alloying with suitable metals [3, 6–10]. Blackman et al [6] studied the applications of binary metal additives in solid propellants and found that the payload capability of propellant systems employing binary metal additives is superior to that containing an equal weight of the denser metal constituent in pure form. Oxidisers like AP can release oxygen and chlorine and are strong oxidizing agents. Combination of metals having an affinity towards these oxidising agents can be used to leverage this possibility of improved specific impulse. Higher heat of formation of oxides and chlorides of metals gives an indication about the preference of metal for the formation of oxides over chlorides or vice versa [6]. Aluminium oxide has a higher heat of formation than magnesium oxide and chloride of magnesium has a higher heat of formation than that of aluminium chloride. So, in the present work, a binary alloy of aluminium and magnesium was chosen as an energetic additive in composite propellants with AF and (HTPB).

Tailored composite particles have been applied to improve aluminium particle ignition and combustion. Hatem Belal et al [11] prepared mechanically activated(MA) aluminium and magnesium powder and laser ignition tests showed that the prepared powder is more reactive than magnalium which has the same Al: Mg mass ratio. The results showed that the MA powder increased the burning rate and this increase reached a maximum at 50% Mg. The MA powder showed lower agglomerate size in comparison to neat aluminium propellant or physical mixture with the same composition of Mg. Breiter et al [3] found that the oxidation rate and ignitability of certain Al–Mg alloys are higher compared to those of Mg. Steady lifted flames of aluminium, magnesium and mechanically alloyed powders were studied by Yuriy L. Shoshin et al [8] and found that the mechanical alloy powder particle of Al and Mg has a combustion time ~2.6 ms which is around 4 times less than the observed combustion time of Al of 10 ms.

Nanopowder of hypo-eutectic Al–Mg alloy was prepared by the electrical wire-explosion technique in the current study. Thin wires of the alloy were cut by electro-discharge machining (EDM) and exploded electrically. The powder is collected and analysed using various techniques for microstructural characterisation. The chamber pressure has a direct relation with the size distribution of condensed nanoparticles [12]. It is observed that the size of the critical nucleus reduces with the reduction in explosion chamber pressure [12–15]. The chamber pressure was set to 25 kPa to make the powder finer. Explosions were carried out to produce powder and the synthesised powder was stored in an airtight container for preventing air oxidation. The synthesised nanopowder is used as an energetic additive in composite solid propellant in the current study.

2. Experimental details

2.1. Production of nanopowder of hypo-eutectic Al–Mg alloy

Aluminium-magnesium (78% and 22% respectively, by weight) alloy castings were prepared and thin wires were cut by electric discharge machining. Nanopowder of Al–Mg alloy was prepared by electrical explosion of thin wire (EEW) and further condensation of the saturated metal vapours in an inert gas atmosphere. EEW is inert...
gas pyrolytic method in which, a thin wire is exploded by sudden discharge of high voltage current through it. A voltage-doubler circuit with the thin wire and capacitor arranged in basic RLC circuit are the main parts of the EEW setup. A detailed description of the production of the nano powder of hypo-eutectic Al–Mg alloy and microstructural characterisation are explained by the author in [12]. The chamber pressure was set to 25 kPa to make the powder finer.

2.2. Preparation of propellant
Propellants were prepared with 14% binder consisting of hydroxyl-terminated polybutadiene (HTPB), toluene diisocyanate (TDI) and dioctyladipate (DOA). The synthesized alloy nanopowder is added to 15% by weight of total propellant. The composition is similar to that reported by Jayaraman et al and Manuprasad et al [4, 16]. The binder is hand-mixed thoroughly and alloy powder is added and mixed again. Once binder and nano-alloy powder became a homogenous mixture, coarse and fine ammonium perchlorate (AP) is added. Hand mixing is continued for sufficient time and then the mixture is transferred to a micromixer for further mixing. It is mixed further in the micromixer which has rotating blades running at lower rpm. The mixer provided with a hot water jacket for effective mixing. A hot water bath at 60 to 70 °C, a thermo-controller and a circulation pump system is used for continuous hot water circulation. This mixture is transferred to a casting cup and kept in the oven for curing at 65 °C. Another set of propellants were cast with micron-sized Al–Mg alloy powder procured from Nanoshel. The density of the propellants prepared was calculated using gas pycnometer using helium gas. Weight of the empty cell and cell with the propellant sample is weighed. Weight of the sample is given as an input parameter to pycnometer and the analysis was started. Figure 1 shows image of strands of propellant cut from the cured propellant. The density of the propellant with nano Al–Mg alloy powder was found to be $1.67 \pm 0.05 \text{ g cc}^{-1}$ and that with micro-alloy powder was found to be $1.65 \pm 0.05 \text{ g cc}^{-1}$.

2.3. Burning rate studies using window bomb setup
Mean burning rate of the burning propellant is studied using combustion photography method. The method involves analysing combustion of the propellant by examining images of the burning propellant extracted from the video captured during the combustion. It is one of the most convenient method to calculate the mean burning rate of solid propellant combustion. Window bomb setup is used to measure the mean burn rate of the propellant. The set-up used in the current study was developed and installed by Omar (2000) at this laboratory [17]. A detailed description about the setup is explained in a paper from our research group by Kathiravan et al in [18]. Figure 2 shows a schematic diagram of the window bomb. It consists of a 40 mm thick stainless steel cylinder with two quartz windows (one for illumination and the other for camera access). The bottom flange has a pedestal to mount the propellant with a provision for hose attachment for pressurising the chamber to required pressure. An exhaust hose with a control valve is connected on the top side of window bomb to control the pressure inside the chamber and to flush out the gas after complete burn out of the propellant. Nitrogen gas is used to pressurise the chamber in the current study. The pressure in the chamber is monitored using a Bourdon gauge with a resolution of 0.5 MPa over the entire pressure range. The exhaust valve is partially vented with nitrogen flow during the test, which helps in controlling the rise of pressure in the chamber and to flush the
window off product gases/soot particles produced as a result of the combustion of the propellant, which could obscure the view of the burning surface. A CCD video camera (Sony make, model XC-75CE) with a resolution of $795 \times 596$ pixels$^2$, which is coupled with a Nikon lens of 60 mm aperture, is used to record the burning of the propellant samples. Neutral density filters with different types are suitably attached in the camera to reduce the intensity of the luminous flame. The filter is chosen based on the intensity of the flame of the burning propellant. A high power xenon lamp is used as an external illumination source. Videos of the burning propellants are captured at a framing rate of 25 fps with an interval of 40 milliseconds between successive frames. Images of the burning propellant are extracted from the video. Distance between two points in the image is measured in pixels using ImageJ software and the pixel value is converted to corresponding distance in microns by multiplying it with magnification factor. A video of a standard graph sheet is captured for calculating the magnification factor before starting burn rate experiments. The number of pixels corresponding to a known distance between two grid lines in the graph sheet is found out. The magnification factor is calculated as,

$\text{Magnification factor} = \frac{\text{known distance in microns}}{\text{number of pixels}}$  \hspace{1cm} (1)

The images are analysed frame by frame to find out the mean burning rate. Leading frame front position is marked in the first frame and location of the flame front in the successive frames are also marked. The pixel distance is multiplied with magnification factor to get the vertical regression of the propellant in microns. The flame front positions are plotted against the time interval between the frames. A straight line is fitted with a correlation coefficient $> 99.9\%$ in the plot of displacement of flame front locations versus time interval. Slope of the graph gives the mean burning rate of the propellant which is calculated as,

$\text{Mean burning rate} = \frac{\text{vertical regression of the burning surface}}{\text{time of burning}}$  \hspace{1cm} (2)

The cured propellant is collected and cut with a sharp surgical blade. The typical sample size is $5 \text{ mm} \times 5 \text{ mm}$ in cross-section and $10–12 \text{ mm}$ in height. A very thin layer of silica gel is applied on lateral sides of the sample to inhibit sidewise burning and the top surface is coated with ignition paste (a mix of 70% fine AP (5–10 $\mu$m) and 30% HTPB) to ensure uniform ignition of top layer of the sample and further steady regression of burning surface. Nichrome wire is bent to ‘U’ shape with the bottom of the ‘U’ touching the top surface of the propellant and connected to the terminals of a DC power supply through rods mounted on the bottom flange. The window bomb is pressurised to the required pressure, CCD camera is kept in recording mode and DC power is supplied. Nichrome wire is heated by Joule-heating which ignites the top surface of the propellant and starts regressing vertically downwards. Video of regressing propellant surface is captured and analysed for
finding the mean burn rate at different chamber pressures. Experiments are repeated at least twice in each pressure level for confirming the consistency of the experiment.

3. Results and discussion

The synthesised nanopowder is characterised using different micro-structure characterisation techniques. X-ray Diffraction (XRD) analysis is performed and compared with XRD of parent alloy (figure 3) to confirm the presence of intermetallic phases as in the parent alloy. Scanning Electron Microscope (SEM) and Transmission Electron Microscope (TEM) analysis were performed to study the structure and morphology of powder particles. Spherical nature of powder particles was confirmed as compared to unorthodox shapes of particles produced by mechanical activation of alloy particles [8, 10, 11]. The diameter of the powder particles was measured using ImageJ software from the TEM images of the powder particles and it was found that the diameter ranges from 20 nm to 95 nm. Figure 4(a) shows the SEM image and 4(b) shows the TEM image of the powder particles produced at 25 kPa chamber pressure. Figure 4(c) shows EDX spectrum of nano powder with peaks corresponding to aluminium, magnesium and minor traces of oxygen. TGA analysis of the powder shows an offset temperature of melting around 460 °C which is closer to the eutectic melting point of the binary alloy system of aluminium and magnesium [12]. The early melting behaviour of alloy nanopowder could be leveraged, in the wake of delayed ignition experienced in aluminised propellants because of the alumina layer surrounding metal aluminium. The XRD of powder heated up to 1300 °C shows major peaks corresponding to aluminium alone, this could be due to magnesium getting selectively melted and vaporised and taking part in gas phase metal combustion [12].

Matlab code is used to analyse the captured videos of burning of propellants to find out the mean burning rate of the propellants. Figures 5(a) and (b) show images of burning propellant extracted from the video, with burning surface and the lowest regressing point marked. Successive frames extracted from the video of propellant burning at a chamber pressure of 9 MPa is shown in figure 6. Lowest regressing point is marked in each frame of burning surface of propellant along a vertical marked through the first point marked in the burning surface. Multiplying corresponding pixel values with the magnification factor will give distance in microns. As mentioned earlier, the time gap between successive frames is 40 ms. A graph is plotted with distance against time with $R^2$ fit of 0.999. The slope of the graph gives the mean burn rate (figure 5(c)).

A graph is plotted with the mean burning rate of the propellants against corresponding chamber pressure as shown in figure 7. Mean burn rate of the propellants containing micron-size and nano-size alloy Al–Mg powder is compared with the propellants containing micron-size Al powder and with those reported in earlier studies [2, 4, 16]. At a pressure level of 8 MPa, mean burn rate of micro-aluminised propellants was found to be 10.03 mm s$^{-1}$. Similar trends were reported by Manuprasad et al [16] having the same composition as the micro-aluminised propellants in the current study. Zhi et al [19] studied combustion properties of composite propellants with 20 wt% of RDX and 5 wt% of Al powder. A mean burn rate of 12.48 mm s$^{-1}$ at 10 MPa pressure was obtained for propellant composition with 5 wt% of general grain aluminium (gAl– with a size of 10 microns) and 11.78 mm s$^{-1}$ for the propellants with 2.5 wt% gAl and 2.5 wt% nano Al. Whereas, the mean burning rate of 22.03 mm s$^{-1}$ was obtained at a pressure level of 9 MPa with nano Al–Mg powder and 20.4 mm s$^{-1}$ with micro Al–Mg powder (figure 7). Mean burn rate of composite propellants with mechanically activated Al–Mg powder...
Figure 4. (a) SEM image, (b) TEM image and (c) EDX of the hypo-eutectic binary Al-Mg alloy powder particles.

Figure 5. (a) And (b): propellant burning at 4 MPa and 8 MPa pressure respectively, and (c) regression of burning surface in vertical direction plotted against time.
is below 20 mm s\(^{-1}\) even at higher pressure above 10 MPa as reported by Hatem Belal \textit{et al}\[11\]. Propellants with Al–Mg alloy powder are burning faster than those with micro Al powder and mechanically activated Al–Mg powder. Sergey Sokolov \textit{et al}\[8\] studied the effects of duration of mechanical activation in combustion characteristics of Al–Mg-based systems and it was found that the mean size of the particles was reduced with long duration of mechanical activation.

Burning rate and combustion heat studies of high energy material (HEM) composition containing ammonium perchlorate, divinyl rubber based on butadiene rubber and plasticized with transformer oil along with mechanically activated Al–Mg powder show that the HEMs containing Al–Mg powder mechanically activated for 7 hrs has higher mean burn rate and combustion heat \[8\]. The higher mean burn rate of the propellant can be attributed to improved combustion characteristics and reduction in the mean size of Al–Mg alloy powder added in the propellant. As reported by the author in \[12\], the early melting of Al–Mg nanopowder inferred from the TGA-DSC analysis, as an indication of selective melting of magnesium providing heat feedback to the burning surface and helping in enhanced pyrolysis of the particulate constituents of the propellant.

4. Combustion of Al–Mg alloy powder

A model proposed by A W Blackman \textit{et al}\[6\] for metal combustion for metals having vaporisation temperature of metal oxides greater than vaporisation temperature of the metal suggests a high-temperature reaction zone which is at vaporisation temperature of metal oxide surrounding the core metal. Molten metal in the core gets
vaporised by the conductive and radiative heat transfer from the high-temperature zone, which is at the vaporisation temperature of the metal oxide and the metal vapours diffuse to the high-temperature zone and gets oxidised. As the difference between vaporisation temperature of the metal and metal oxide increases, diffusion of oxygen to the high-temperature zone increases due to the greater diffusional area [6]. This will lead to improved oxidation of metal vapour at the high-temperature reaction zone table 1 shows the melting point of metals and the corresponding oxides of the metals. Clearly from the table, there is an appreciable difference between the boiling point of metals and oxides, which enables improved oxidation and burning of metals.

Possibility of micro-explosions in metal/alloy combustion as observed in miscible fuel mixtures and water/oil emulsions was suggested by Breiter et al [3] and found that it reaches a maximum at the eutectic composition of the binary alloy system. They studied combustion of individual Al–Mg alloy particles in the flame of an oxidiser–fuel flame. The combustion was recorded using motion-picture camera and they observed that, the width of the tracks left by the burning particle in the images of particle combustion, which indicates predominant vapour phase combustion. As magnesium content increases, track width becomes greater which indicates improved vapour phase reaction and reduction in particle burning time. The oxide layer formed around the metal particles by oxidation has an important role in metal combustion. The oxide layer over the Al–Mg alloy particles consists of a mixture of double oxide of MgO, Al₂O₃. As magnesium content in the alloy increases above 1 wt%, distribution of metal oxide over particle become mainly of MgO and can be attributed to surface-active magnesium in the liquid state in the high-temperature reaction zone [3]. A monotonic decrease in the combustion time of particles of alloys with decreasing particle size and, as a rule, with increasing magnesium content was revealed by studies of Zenin et al [20]. Kuwahara et al [21] reported that the burning rate of the solid propellant is closely related to heat feedback from the gas phase to the propellant burning surface where the unreacted constituent elements are getting heated and taking part in pyrolysis.

Quijano et al [7] observed emission pulses with short duration of pulses in alloy powders with the higher magnesium content. Also, with increased content of Mg, burn time decreases with a reduction in particle size and burn time is less than pure Al and Mg powder of the similar particle size. Chen et al [10] observed that the burning time of mechanically alloyed Al–Mg alloy particles in a CH₄–air premixed flame established on a Meeker burner is linearly dependent on particle size. Experiments by Huang et al [22] revealed that propellant samples containing ball-milled Al–Mg alloy powders exhibited good performance in terms of the burning rate, primary combustion heat, and Al reaction efficiency compared to that of the propellant samples containing Al powders. Shorter burn time observed for higher Mg content could be an indication of evaporation of Mg and its reaction in the vapour phase, which accelerates the heat feedback and contributes to enhancing the reaction kinetics of Al.

It is conclusive from various previous studies that heat feedback from vapour phase reaction is conducive in improving the burning rate of propellants. The inclusion of synthesized nanopowder of Al–Mg alloy in propellants leverages the advantage of reduced size and increased heat feedback for an improved mean burning rate of the propellant. The increase in the mean burning rate obtained with the propellants containing the synthesized nanopowder of Al–Mg alloy in the current study could be explained in the lights of above-mentioned points.

5. Conclusion

- Nanopowder of hypo-eutectic Al–Mg alloy was prepared by electrical explosion method.
- Spherical size of powder particle was confirmed by SEM and particle size was measured using TEM images.
- Composite propellants were prepared with bimodal AP and nano, micro-alloy Al–Mg powder and micro Al powder.
- Mean burn rate was found out using window bomb setup at a chamber pressure of 1 MPa to 9 MPa with an incremental step of 1 MPa.
• Mean burn rate of 22.03 mm s$^{-1}$ was obtained at a pressure level of 9 MPa with nano Al–Mg powder and 20.4 mm s$^{-1}$ with micro Al–Mg powder compared to mean burning rate of 10.03 mm s$^{-1}$ at 8 MPa pressure for aluminised propellants.

• Early combustion of magnesium provides heat feedback to the high-temperature reaction zone and thus augments the reaction of aluminium.

• Presence of low melting eutectic composition and selective melting of magnesium from the alloy and taking part in combustion reaction in vapour phase could be attributed to the improved burning rate of the propellants with Al–Mg alloy powder.

• Al–Mg nano and micro powder could be used as a potential alternative to aluminium powder as an energetic additive in solid propellants as evident from the present study and a detailed study on agglomerates and two-phase flow loss forms a future scope of the work.

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